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INSENSITIVE MUNITIONS AND ENERGETIC MATERIALS (IMEM) TECHNOLOGY SYMPOSIUM

Enhancing Performance of Insensitive Munitions

April 7 – 8, 2021 | NDIA.org/IMEM21

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NDIA

WHO WE ARE

The National Defense Industrial Association is the trusted leader in defense and national security associations. As a 501(c)(3) corporate and individual membership association, NDIA engages thoughtful and innovative leaders to exchange ideas, information, and capabilities that lead to the development of the best policies, practices, products, and technologies to ensure the safety and security of our nation. NDIA's membership embodies the full spectrum of corporate, government, academic, and individual stakeholders who form a vigorous, responsive, and collaborative community in support of defense and national security. For more than 100 years, NDIA and its predecessor organizations have been at the heart of the mission by dedicating their time, expertise, and energy to ensuring our warfighters have the best training, equipment, and support. For more information, visit **NDIA.org**

WELCOME TO THE 2021 VIRTUAL INSENSITIVE MUNITIONS & ENERGETIC MATERIALS TECHNOLOGY SYMPOSIUM

On behalf of the IMEM Committee, I would like to welcome you to the 2021 Virtual Insensitive Munitions and Energetic Materials Technology Symposium. This international gathering of the top chemists, system designers, and engineers from government laboratories, private industry, and academia provides a forum for the exchange and dissemination of cutting-edge research in synthesis, formulation, system design, testing, characterization, and safety—all aimed at both advancing munitions effectiveness and improving safety for the warfighter.

Advancements made by the scientists and engineers presenting at this symposium are directly responsible for the development and fielding of higher-performing, safer munitions; however, challenges remain. It is through the continuing research, development, and collaboration of the members of this community and across our international defense industry that these challenges will be overcome.

Melissa Hobbs-Hendrickson
Senior Director, Business Development,
Elbit Systems of America

Chair, Insensitive Munitions & Energetic
Materials Committee, Munitions Division, NDIA

EVENT INFORMATION

EVENT WEBSITE

NDIA.org/IMEM21

SURVEY AND PARTICIPANT LIST

You will receive via email a survey and list of participants (name and organization) after the conference. Please complete the survey to make our event even more successful in the future.

EVENT CONTACTS

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SPEAKER GIFTS

In lieu of speaker gifts, a donation is being made to the Fisher House Foundation.

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AGENDA

WEDNESDAY, APRIL 7

9:00 – 9:15 am

*All times are in Eastern Daylight Time (EDT)

WELCOME & INTRODUCTION

Gen Hawk Carlisle, USAF (Ret)
President and Chief Executive Officer, National Defense Industrial Association (NDIA)

9:15 – 10:00 am

JOINT ENHANCED MUNITIONS TECHNOLOGY PROGRAM UPDATE

Lawrence Fan
Program Manager (Acting), Joint Enhanced Munitions Technology Program, Office of the Under Secretary of Defense for Research & Engineering

10:00 – 10:15 am

NETWORKING BREAK

ENERGETIC MATERIALS	POLICY
Session Chair: Steve Nicolich	Session Chair: Tom Swierk

10:15 – 10:35 am

TNT Exudation and Crystal Growth
Dr. Ernest Baker
Technology Specialist Officer, Warheads Technology, Munitions Safety Information Analysis Center (MSIAC), NATO

NATO AC326 Subgroup B Ammunition Systems Design and Assessment – Insensitive Munitions Test STANAG Updates, Part 2
Ken Tomasello
Program Manager, Insensitive Munition Advanced Development, Naval Ordnance Safety & Security Activity

10:35 – 10:55 am

Characterization of Nano Octol Formulation
Dr. Daniel Iwaniuk
Chemist, Explosives Development, Armaments Center, U.S. Army Combat Capabilities Development Command

Revisions and Improvements to the NATO Insensitive Munitions Test Doctrine Portfolio
Daniel Pudlak
Principal Investigator, Armaments Center, U.S. Army Combat Capabilities Development Command

10:55 – 11:10 am

NETWORKING BREAK

PROCESSING	TESTING
Session Chair: Dr. Ernest Baker	Session Chair: Ken Graham

11:10 – 11:30 am

Improved Particle Size Reduction & Evaluation Technology for Energetics at HSAAP
Dr. Jacob Morris
R&D Manager, BAE Systems

Variability of Munition Response to Operational and Peace Time Threats
Martijn van der Voort
Technical Specialist Officer, Munitions Transport & Storage Safety, MSIAC, NATO

11:30 – 11:50 am

Influence of Ageing on the IM Signature of an Anti-Aircraft Missile Propulsion Unit

Quentin Weisse
Military Engineer, Expertise Program,
DGA Missiles Testing (French MoD)

Hervé Benard
Head, DGA Missile Testing Expertise Division
(French MoD); Munition Officer, MMCM Program, OCCAR

11:50 am – 12:05 pm

NETWORKING BREAK

SYNTHESIS	POLICY
Session Chair: Andrew Wilson	Session Chair: Christelle Collett

12:05 – 12:25 pm

Development and Testing of NTFA

Dr. Sarah Headrick
Senior Principal Scientist II, BAE Systems

Harmonisation of NATO Insensitive Munitions & Hazard Classification Standards and Practices

Matthew Ferran
Technical Specialist Officer, Munitions Systems, MSIAC, NATO

12:25 – 12:45 pm

Continuous Reactors for Energetic Materials Synthesis

Dr. Joseph Rheinhardt
Research Chemist, CS Squared LLC

Defects Taxonomy & Lexicon

Matthew Ferran
Technical Specialist Officer, Munitions Systems, MSIAC, NATO

12:45 – 1:05 pm

Quantitative Structure-Property Relationship Models for Secondary Explosive Compounds

Carson Britt
Research Chemist, CS Squared LLC

Risk Associated with Under Classification of Small Arms Ammunition

Christelle Collett
Technical Specialist Officer, Propulsion Technology, MSIAC, NATO

1:05 – 1:20 pm

CLOSING REMARKS

Melissa Hobbs-Hendrickson
Senior Director, Business Development, Elbit Systems of America
Chair, Insensitive Munitions & Energetic Materials Committee, Munitions Division, NDIA

THURSDAY, APRIL 8

9:00 – 9:05 am

OPENING REMARKS

Ron Hollands
Chief Materials Technologist, BAE Systems

9:05 – 9:20 am

MSIAC – HIGHLIGHTS AND FUTURE PRIORITIES

Charles Denham
Project Manager, MSIAC, NATO



NDIA
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64TH ANNUAL FUZE CONFERENCE

Continued Fuze Advancements amid a Global Pandemic

Register Today

In 2021, the Fuze Section of NDIA's Munitions Technology Division aims to crack the code of fuze advancements—including their development, production, and performance—in relation to defense and national security within the time of a global pandemic. Accordingly, this event will virtually convene stakeholders from industry, government, and academia to advance the field and art of fuzing for the benefit of our current and future warfighters. Over the course of two days, attendees will network with fellow defense professionals from around the world, participate in engaging sessions, and explore the transformation of the theoretical into the practical. With its packed agenda and focused mission, this event is not to be missed. Secure your virtual spot online today.

May 11 – 12 | NDIA.org/Fuze21

9:20 – 9:30 am

MUNITION SAFETY AWARDS CEREMONY

Charles Denham
Project Manager, MSIAC, NATO
Award Presenter

ENERGETIC MATERIALS Session Chair: Mike Ervin	MODELING Session Chair: Wade Babcock

9:30 – 9:50 am

Evaluation of LX-14 Using FEM HMX

John Latwinas
Chemical Engineer, Armaments Center,
U.S. Army Combat Capabilities Development Command

Application of Herd Immunity to Munitions Safety

Martijn van der Voort
Technical Specialist Officer, Munitions Transport & Storage Safety, MSIAC, NATO

9:50 – 10:10 am

Evaluation of PBXN-9 Utilizing FEM HMX

Dr. Daniel Iwaniuk
Chemist, Explosives Development, Armaments Center,
U.S. Army Combat Capabilities Development Command

Modeling of the M795 155 mm Artillery Projectile Sympathetic Reaction Test

Dr. Erik Tolmachoff
Senior Engineer, CS Squared LLC

10:10 – 10:25 am

NETWORKING BREAK

ENERGETIC MATERIALS Session Chair: Paul Braithwaite	TESTING Session Chair: Ken Graham

10:25 – 10:45 am

Novel Recrystallization Procedure to Generate High Bulk Density NQ (HBDNQ) with an Average Particle Size of Less Than 50 µm

Dr. Tomasz Modzelewski
R&D Chemist, BAE Systems

Influence of Mechanical Properties on the Explosiveness of Energetic Materials

Morgan Bolton
Energetic Material Scientist, Defense Ordnance Safety Group, ST1

10:45 – 11:05 am

Advancing Particle Size Characterization Technology: Dynamic Image Analysis

Dr. Teresa Kirchner
Principal Scientist, BAE Systems

11:05 – 11:20 am

NETWORKING BREAK

ENERGETIC MATERIALS Session Chair: Melissa Mileham	TESTING Session Chair: Ron Hollands

11:20 – 11:40 am

An International Review on Green Insensitive Plastic Explosives

Christelle Collett
Technical Specialist Officer, Propulsion Technology, MSIAC, NATO

Re-Development & Manufacture of LX-04 Explosive at HSAAP

Brian Alexander
R&D Senior Principal Chemist and R&D Safety Officer, BAE Systems

11:40 am – 12:00 pm

Repurposing IMX-101 Risers for Use in the Mining, Oil, and Gas Industries

Radhika Kasabwala
Mechanical Engineer, Explosives Department Branch, Armaments Center, U.S. Army Combat Capabilities Development Command

Process Improvement of High Performance Explosive PAX-2A

Virgil Fung
R&D Senior Principal Scientist and Program Manager, BAE Systems

12:00 – 12:15 pm

NETWORKING BREAK

ENERGETIC MATERIALS	PROCESSING
Session Chair: Jamie Neidert	Session Chair: Melissa Hobbs-Hendrickson

12:15 – 12:35 pm

Nanomaterials as Energetic Fillers

Christopher Hollands
Technology Specialist Officer, Energetic Materials, MSIAC, NATO

Artillery Projectile Prematures Historical Review

Dr. Ernest Baker
Technology Specialist Officer, Warheads Technology, MSIAC, NATO

12:35 – 12:55 pm

Advanced High-Performance Minimum Smoke Propellants

Cameron Lee
Senior Engineer, Energetics and Propellant Development Group, Aerojet Rocketdyne

IM Upgrade of the Italian Army's 155 mm Artillery Projectiles

Dr. James Padfield
Head, Explosives Engineering, RWM Italia SpA

12:55 – 1:15 pm

Advanced Insensitive Munitions & Energetic Materials Concepts Using 1.5-Nanometer Titanates & Zirconates

Salvatore Monte
President, Kenrich Petrochemicals, Inc.

Barrel Heating for Hot Gun Cook-Off Thermal Analysis

Dr. Jon Yagla
Bowhead Technical Services

1:15 – 1:35 pm

Allied Ordnance Publication (AOP-7) Revision Status

Ken Tomasello
Program Manager, Insensitive Munition Advanced Development, Naval Ordnance Safety & Security Activity

A Review of Mitigation Techniques for Small Calibre Munitions and Flares

Christelle Collett
Technical Specialist Officer, Propulsion Technology, MSIAC, NATO

1:35 – 1:45 pm

CLOSING REMARKS

Gen Hawk Carlisle, USAF (Ret)
President and Chief Executive Officer, NDIA

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KEYNOTE BIOGRAPHY



LAWRENCE FAN

Program Manager (Acting), Joint Enhanced Munitions Technology Program
Office of the Under Secretary of Defense for Research & Engineering

Mr. Lawrence Fan is a program manager for DoD fuzing and munitions technology and development programs. Mr. Fan is based out of the Energetics Technology Department at the Naval Surface Warfare Center Indian Head Division. Since entering government service in 1990, he has supported fuzing development for gun projectile, mine clearance and torpedo applications. He has headed several fuze R&D projects including the development of the S&A Device for the Navy's Countermeasure Anti-Torpedo Torpedo. Since 2010, Mr. Fan has served as the program manager for the OUSD Research and Engineering (R&E) Joint Fuze Technology Program (JFTP). In 2020, Mr. Fan assumed the Program Manager (Acting) duties for the OUSD R&E Joint Enhanced Munitions Technology Program (JEMTP). The JFTP and JEMTP invests and executes 6.2 and 6.3 munitions technology projects with a focus on pervasive DoD needs. Mr. Fan is also the Navy lead in the DoD Fuze IPT which supports Department fuzing R&D, acquisition, policy and industrial base issues.

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U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Characterization of Nano Octol Formulation

Presenter: Daniel Iwaniuk (daniel.p.iwaniuk.civ@mail.mil)

Principal Investigator: Philip Samuels (philip.j.samuels2.civ@mail.mil)

Team: Dr. Hongwei Qiu (Leidos), Mr. Erik Wrobel, Dr. Henry Grau, Dr. Victor Stepanov, Dr. Rajen Patel, Dr. Reddy Damavarapu, Dr. Kelley Caflin, Mr. Joshua Felts (Indian Head)

DEVCOM AC; Picatinny Arsenal, NJ

Distribution A

BACKGROUND

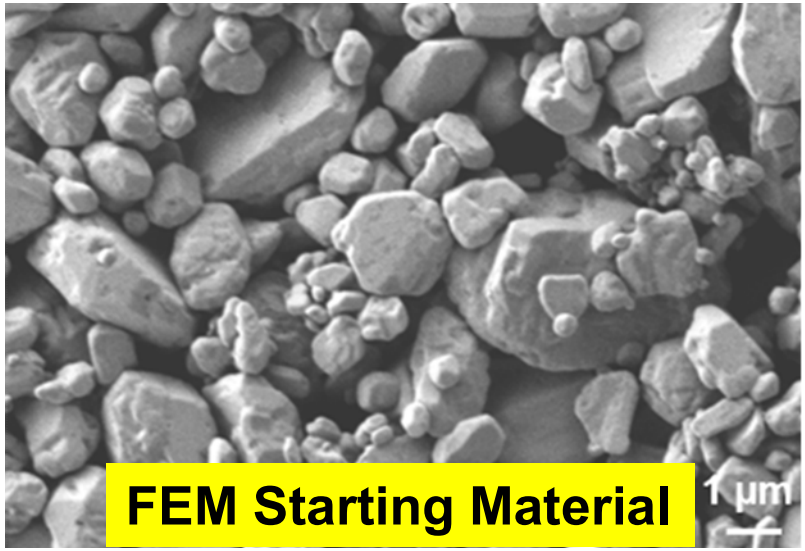
- Over the last several years, DEVCOM AC engineers have demonstrated the manufacturing capability of producing and uniformly coating nano-energetics

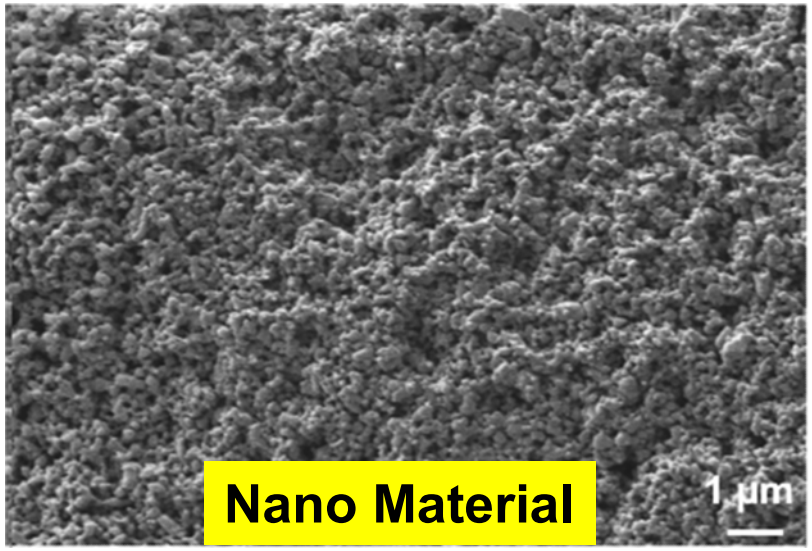
- The use of nanoscale high explosives (HEs) along with a uniform polymeric coating effectively eliminates large voids that are commonly regarded as the main locations for the formation of hot-spots and detonation initiation upon unplanned external stimuli.

- Improved mechanical properties of nano-energetic based explosives may also help with setback survivability.

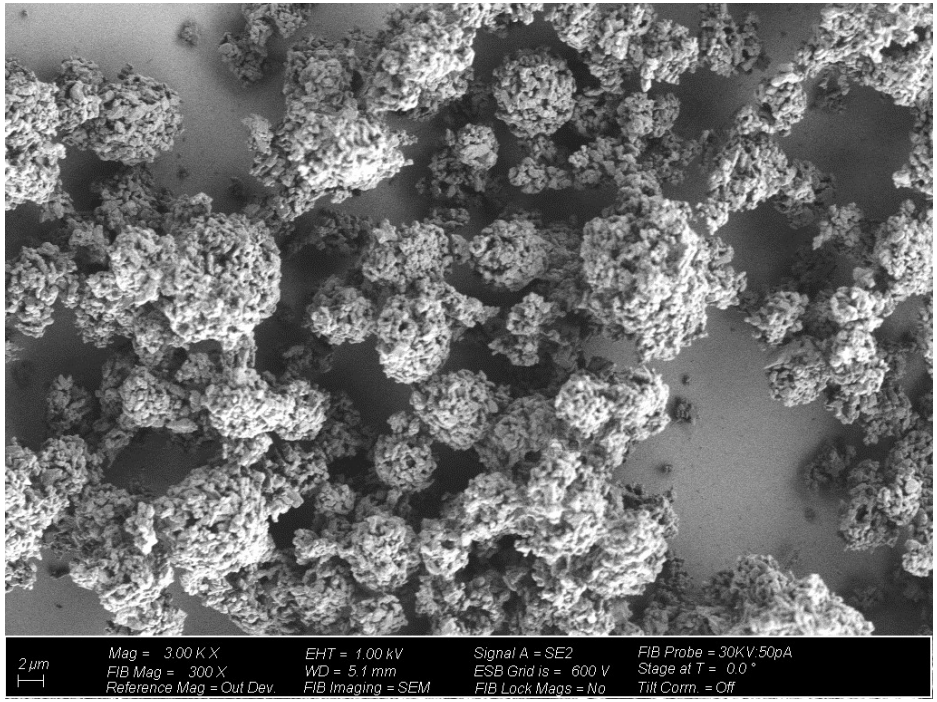
- The majority of the explored formulations utilizing nanoscale HEs were based on polymer-bonded explosives.

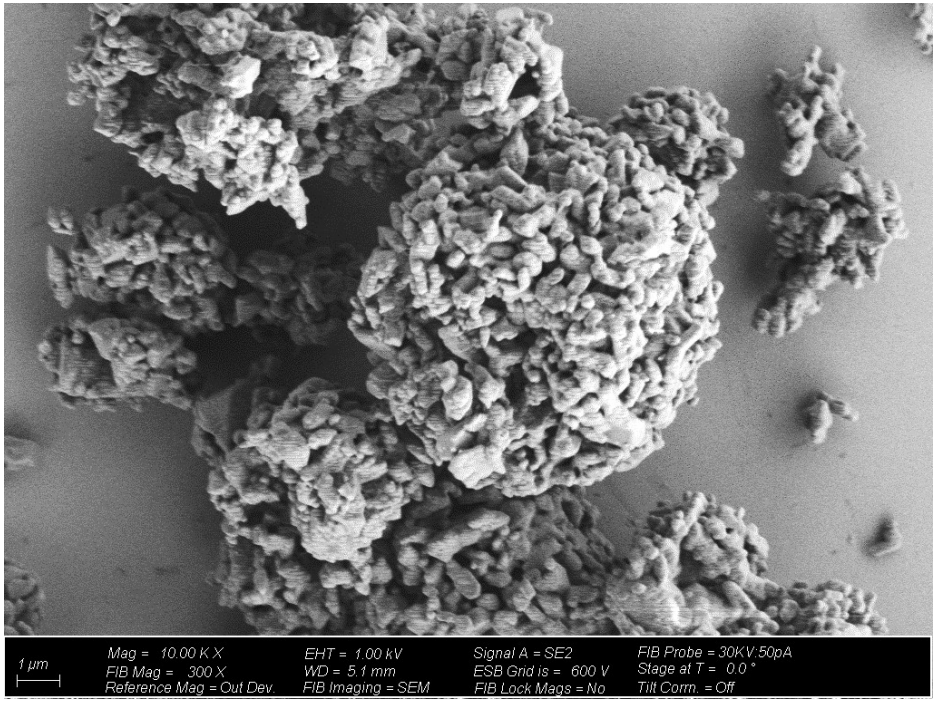
TECHNICAL APPROACH





SEM IMAGES OF NANO OCTOL



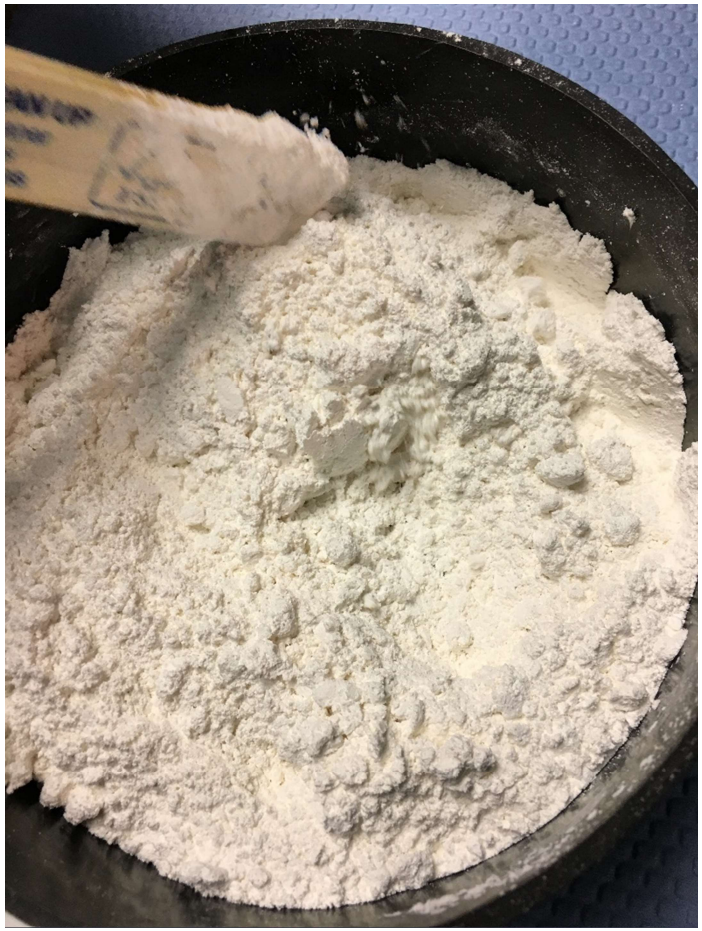


JAGUAR CALCULATIONS

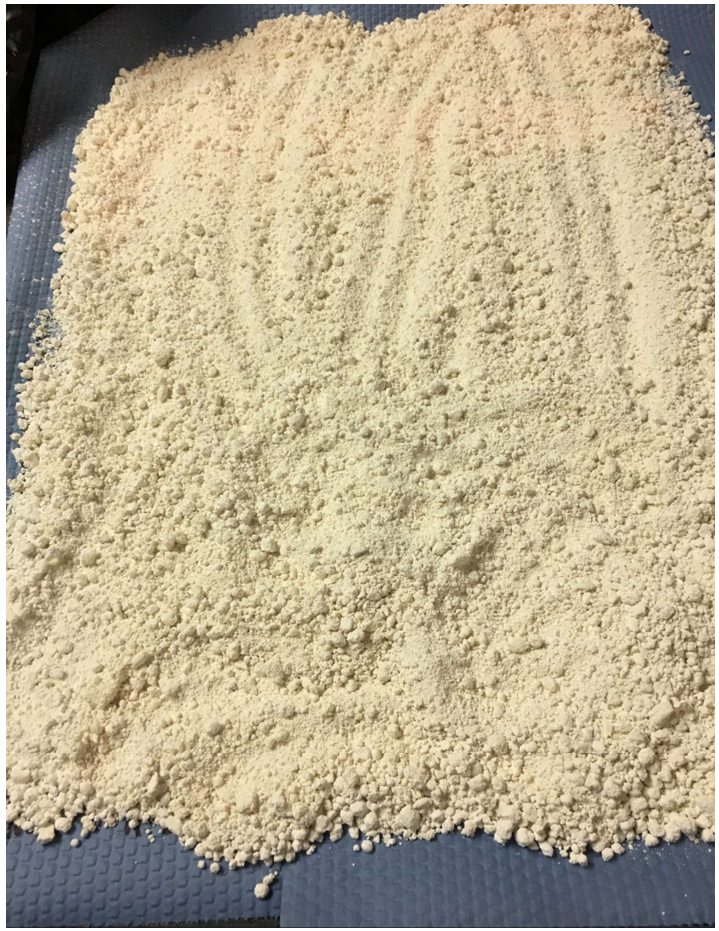
- Jaguar Version 18 Thermo-chemical code was utilized for conducting legacy formulations compared to Nano Octol at 98% TMD
- Nano Octol calculations show equivalent or better and Octol 70:30 and PBXN-9

Formulation	Density (g/cc)	DV (km/s)	CJ Pressure (kbar)	2 Volume Expansions	7 Volume Expansions
Octol (70:30)	1.786	8.24	30.3	2.64	2.87
Octol (75:25)	1.799	8.36	31.2	2.66	2.90
PBXN-9	1.749	8.28	29.6	2.64	2.87
PBXN-5	1.864	8.73	34.7	2.74	2.97
LX-14	1.817	8.62	33.4	2.7	2.94
Nano Octol	1.781	8.32	30.6	2.68	2.88

GRANULATION OF NANO MOLDING POWDER



Nano Octol granulated with Water



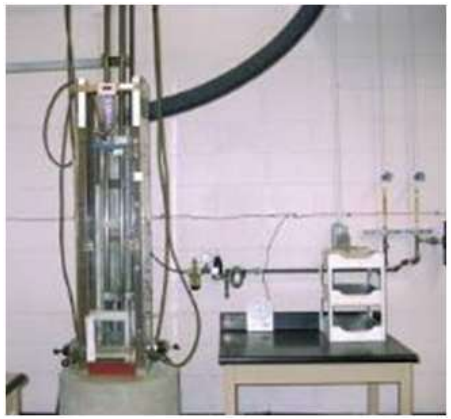
Nano Octol granulation

Increasing the bulk density of the powder, not increasing the PSD of the crystals since they are already coated

Bulk Density: ~ 0.8 g/cc

SAFETY TEST RESULTS

Formulation	ERL Impact (cm)	BAM Friction (N)	ABL ESD (J)
FEM HMX	56.2	0/10 Rxn @ 144	0/20 Rxn @ 0.063
Octol (70:30)	39	0/10 Rxn @ 252	0/20 Rxn @ 0.020
Nano Octol	No Rxn in 10 trials @ 100	0/10 Rxn @ 288	0/20 Rxn @ 0.016
RDX Class I	26.1	0/10 Rxn @ 160	0/20 Rxn @ 0.031
HMX Class I	32.8	0/10 Rxn @ 108	0/20 Rxn @ 0.031



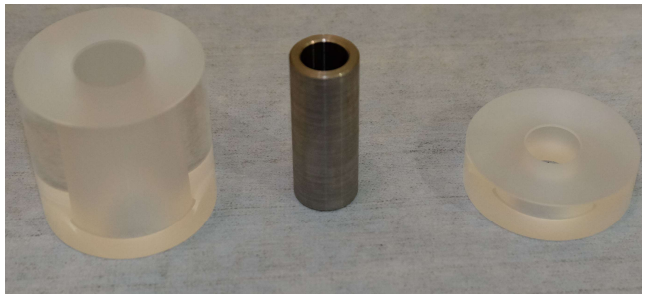


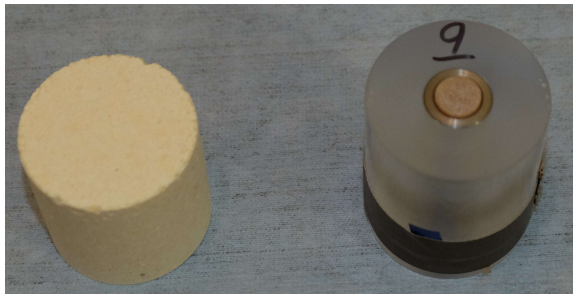


IHE GAP TEST RESULTS SUMMARY

- The IHE Gap test was executed per US National Section of AOP-7 (Draft SRD-AOP-7.2) which uses a pressed pentolite explosive (density of 1.56 g/cm^3) donor system
- This consists of either two stacked pellets or one pellet (2.0 inches thick and 2.0 inches in diameter), each 1.0-inch thick and 2.0 inches in diameter
- Polymethylmethacrylate (PMMA) spacers are used as the attenuator material between the donor and acceptor charges.
- Utilized MSIAC Newgates for IHE Card Gap pressure conversion

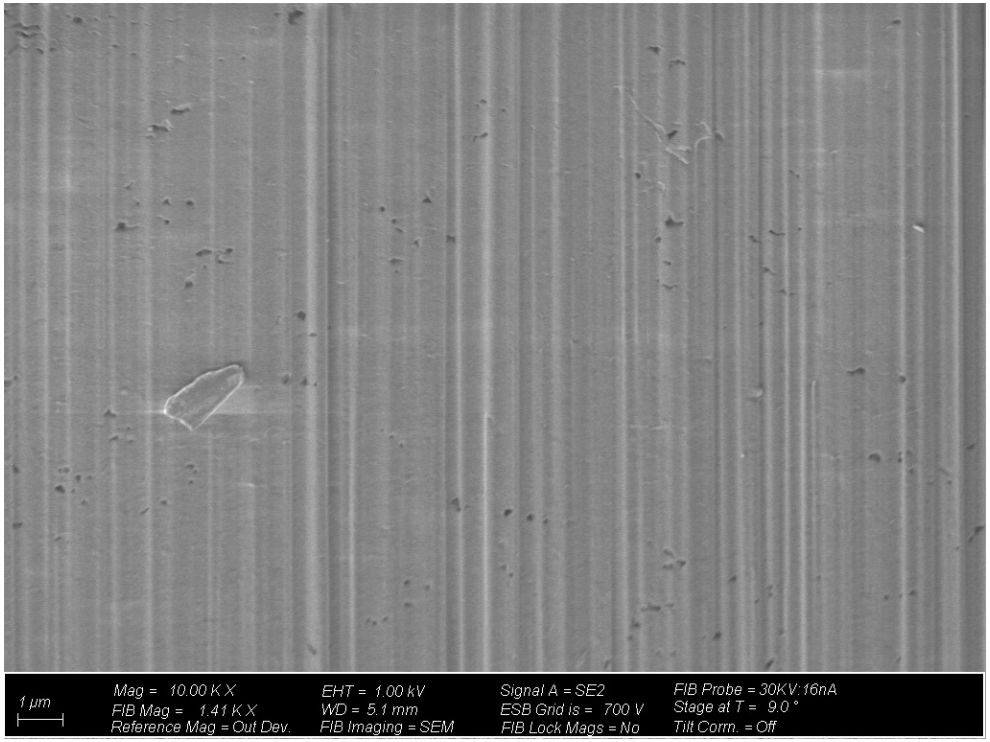
Formulation	Density (g/cc)	Cards
Comp B (Melt Pour)	1.7	175.5
Nano Comp B	1.71	Noted Improvement
Octol 70:30 (Melt Pour)	1.77	172-180
Nano Octol	**1.76**	**Noted Improvement**

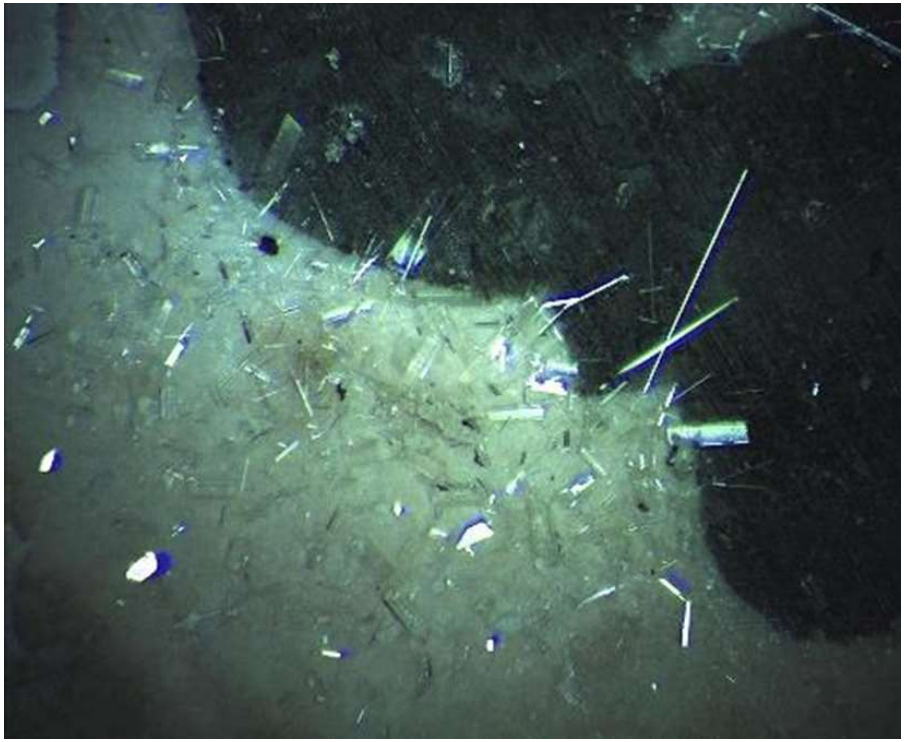






FOCUSED ION BEAM-SEM IHE GAP PELLET





- No large internal voids observed
- However, TNT crystal growth was observed from the top of the Nano Octol IHE Gap test pellets
- Crystal growth might lead to the consolidation of voids, resulted in the increase of shock sensitivity

DETONATION VELOCITY / CJ PRESSURE

Explosive	Density (g/cc)	DV (km/s)	CJ Pressure (kbars)
LX-14	1.801	8.74	337.6
PBXN-9	1.740	8.52	297 - 318
Octol 70:30	1.795	8.35	303
Nano Octol	**1.765**	**8.42**	**304**

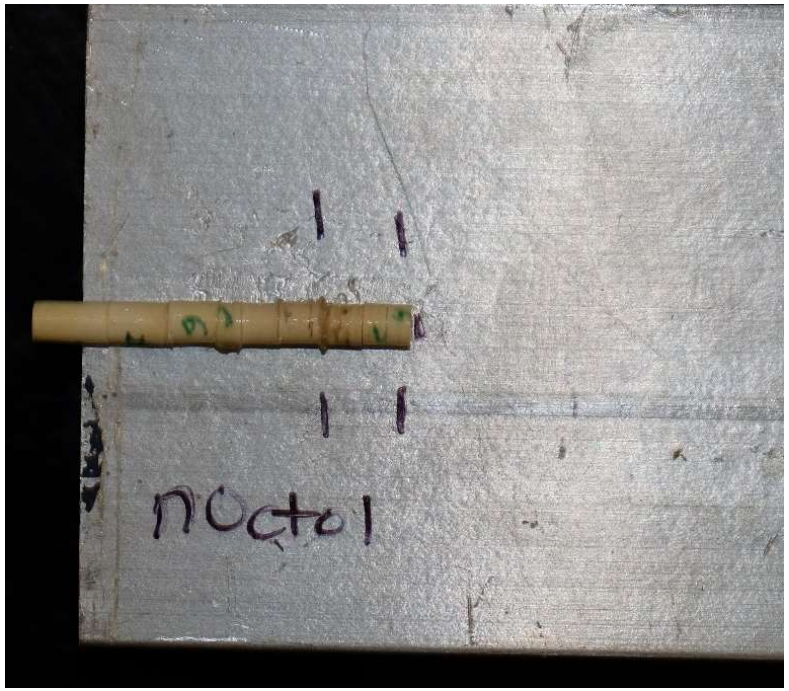
- Nano Octol were tested in 0.5” Diameter Detonation Velocity with calibrated witness plates
  - Used 0.5” x 0.5” Comp A-3 Type II booster initiated by RP-502 EBW
- LX-14 and PBXN-9 CJ Pressures measured by aquarium test
- Octol 70:30 CJ Pressure measured by PDV

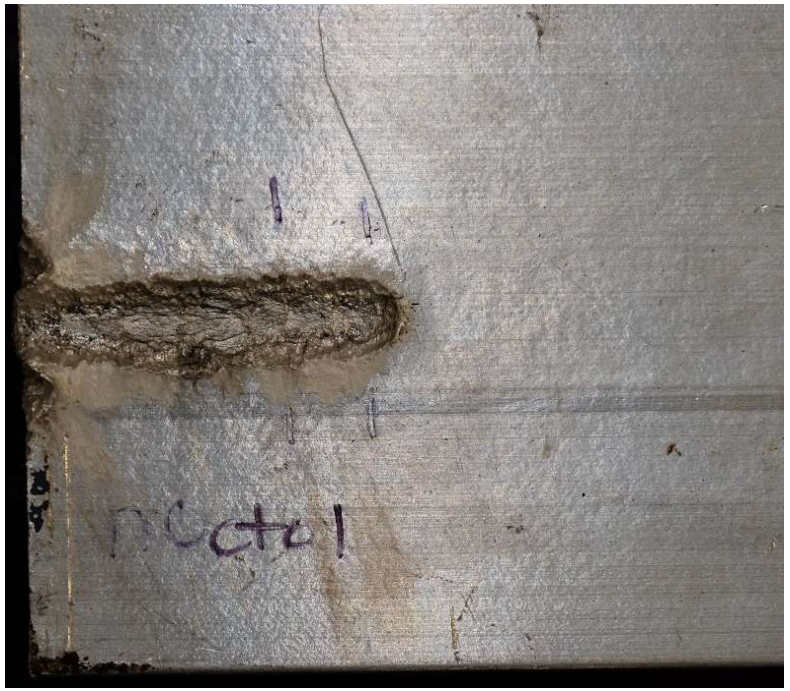




CRITICAL DIAMETER

- Nano Octol pressed to 1.72 g/cc detonated at 0.125” diameter
- Smaller pellets were not tested due to not being able to stack the pellets (too small and brittle)
- Pellets were placed on their side on an aluminum plate, greased, and aligned
- Full stack detonated





TECHNICAL CONCLUSIONS

- Nano Octol maintained performance level of PBXN-9 and Octol 70:30
  - DV/CJ Pressure: 8.42 km/s / 304 kbar

- Nano Octol demonstrated small critical diameter
  - Detonated at 0.125” diameter (density: 1.72 g/cc)

ACKNOWLEDGEMENTS & TEAM MEMBERS

CCDC AC

Erik Wrobel
Joel Rivera
Garrett Richards
Gerard Gillen
Mike Van De Wal
Ralph Acevedo
Jason Semanek
Dr. Dan Iwaniuk
Dr. Henry Grau
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Rob Decker
Dr. Rajen Patel
Dr. Victor Stepanov
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Dr. Reddy Damavarapu

Indian Head

Joshua Felts

Leidos

Dr. Hongwei Qiu
Dr. Raja Duddu

QUESTIONS?

NATO AC/326 SG/B Ammunition Systems Design and Assessment

Insensitive Munitions Test STANAG Updates (Part 2)

Ken Tomasello

Chairman AC/326
SG/B

Distribution Statement A: Approved for public release; distribution is unlimited

Acknowledgements

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Michael Sharp

Defence Science & Technology Edinburgh (AUS DoD), Adelaide, Australia

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Outline

- BLUF
- Background
- NATO Documentation Policy
- IM Standards Hierarchy
- AOP-39 Update (Edition D)
- SRD AOP-39.1
- Status of IM Test Standards
- IM Test Procedure AOP Version Update
- Future IM/HC Harmonization
- Concluding Remarks

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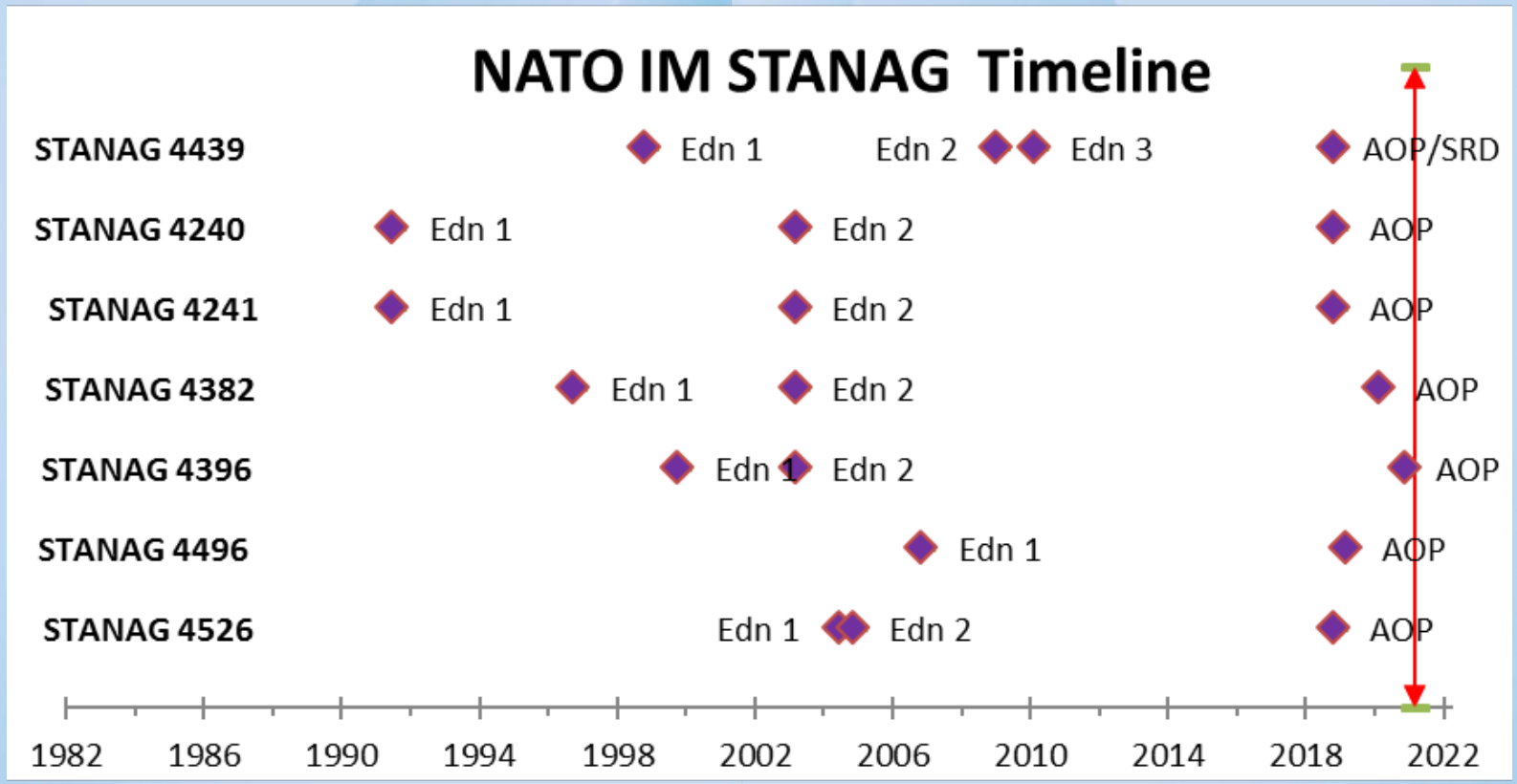
BLUF

- During the past five years all seven of the NATO Insensitive Munitions (IM) test STANAGs and Allied Publications (AP) have been updated.
- Each document has been updated to the current NATO standardization requirements spelled out in AAP-03K.
- The overarching IM test documents STANAG 4439 and AOP-39 (*"Policy on the Development and Assessment of IM"*) were part of the update.
- The guidance provided by the updated standards will ensure more consistent testing and evaluation of ordnance hazard assessment testing.

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Background

- Last coordinated publication of IM Test STANAGs April 2003
- STANAG 4439 / AOP-39 revised twice since last Test STANAG update
  - Resulting inconsistencies
- Changing organisation and structures
- Opportunity with transition of Test STANAGs to AOPs

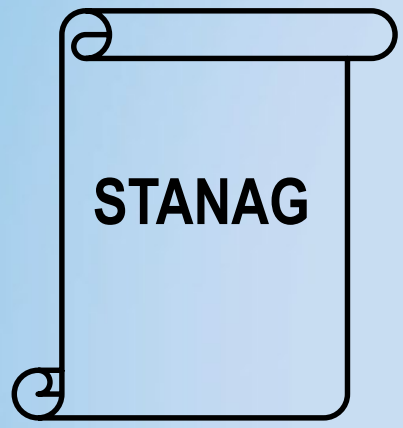


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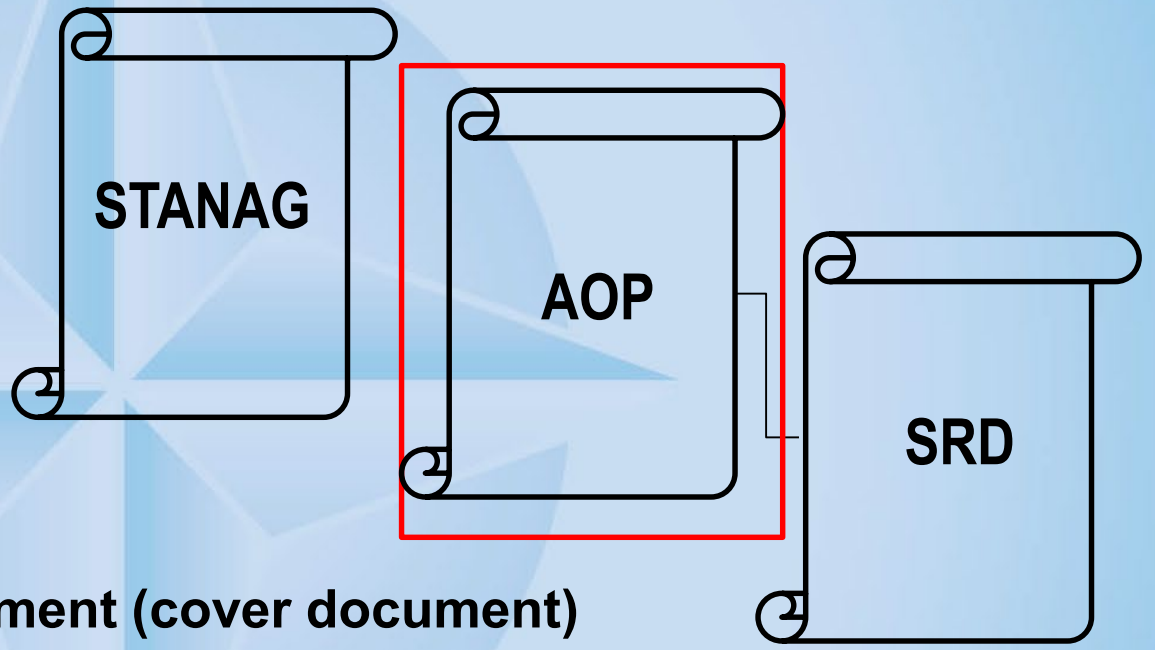
NATO Documentation Policy

NATO Policy on document structure: AAP-03(K)

Before



Now



- **STANAG : STANdardization AGreement (cover document)**
- **AOP : Allied Ordnance Publication (technical document)**
- **SRD : Standard Related Document (recommendations)**

Differences in ratification and promulgation:

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NATO Documentation Policy

Standardization Agreement (STANAG)

- *"…a NATO standardization document that specifies the agreement of member nations to implement a standard… in order to meet an interoperability requirement*."
- No technical content.
- Has to be ratified by a minimum number of nations

Allied Publication (AP)

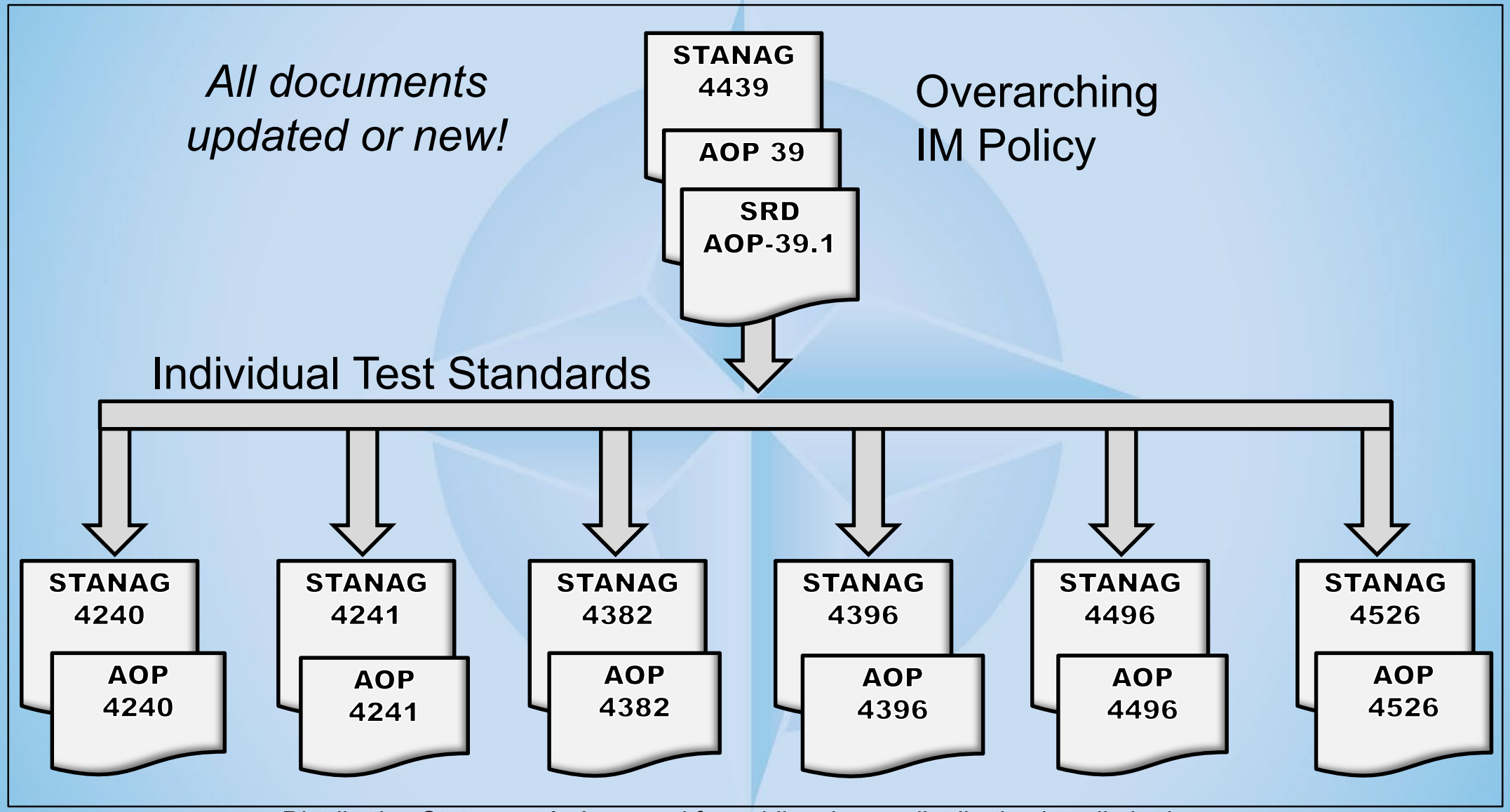
- Technical content – policy, requirements etc.
- Promulgated once covering STANAG has been ratified
- Change process:
  - Minor changes – Version Update (Ed. A V1 ➔ Ed. A V2) – update of STANAG not reqd
  - Major changes – Edition Update (Ed. A V1 ➔ Ed. B V1) – STANAG requires update & ratification

Standards-Related Document (SRD)

- *"…a NATO standardization document that facilitates understanding and implementation of one or more Allied standards"*
- Provides additional data and information, but no policy or requirements (e.g. manuals, catalogues)
- All changes approved by silence procedure – not update to STANAG required.

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IM Standards Hierarchy



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AOP-39 Update (Edition D)

- Two strands of work for AOP-39 update
  - Review of Test Conduct and Reporting (Annex H)
  - Review of IM Response Descriptors (RD) (Annex I)
- Annex H: Largely removed
  - Common aspects of conduct and documentation in SRD
  - Technical aspects in individual test AOPs
  - Test specific conduct and reporting in individual test AOPs
  - NB remaining elements now superseded by publication of 6 full-scale AOPs – Annex H will be removed from next Edition of AOP-39
- Annex I
  - IM Response Descriptors updated
    - Fragment with energy level > 20 J at 15 m, regarding its mass and nature: Steel, Aluminum, Tungsten and HDPE
  - Articles Under Test more defined and prescribed
  - Calibration Testing promoted and more fully prescribed

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SRD AOP-39.1 (Edition A)

- Common aspects of conduct and documentation
  - Test framework, organization and responsibilities
  - Test program
  - Test planning and guidance
  - Test specific considerations
  - Documenting
  - Test conduct
  - Reporting

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Current Status of Test Standards

Standard	Title	Edition	Date
STANAG / AOP-4240	Fast Heating Munition Test Procedures	Ed A V1	NOV 2018
STANAG / AOP-4241	Bullet Impact Munition Test Procedures	Ed A V1	NOV 2018
STANAG / AOP-4382	Slow Heating Test Procedures for Munitions	Ed A V1	MAR 2020
STANAG / AOP-4396	Sympathetic Reaction Test Procedures for Munitions	Ed A V1	DEC 2020
STANAG / AOP-4496	Fragment Impact Test Procedures for Munitions	Ed A V1	MAR 2019
STANAG / AOP-4526	Shaped Charge Jet Munition Test Procedure	Ed A V1	NOV 2018

All available on website of NATO Standardization Office: https://nso.nato.int/

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AOP-4240: Fast Heating

- Test Methods – *Modified*
  - Test Method 1: Large pool fire
  - Test Method 2: Mini pool fire
  - Test Method 3: Fuel burner fire **NEW**
    - Characterized by average heat flux > 80 kW/m^2 over 30 s period once 800 °C is reached
- Themocouples – *Modified*
  - Minimum 6 TC: (40-60 mm) fore, aft, starboard, port, above and below
- Conformity – *Modified*
  - Taverage > 800 °C measured by all TC
  - 550°C under 30 s measured by all TC





US NSWCDD 3.7 m square propane burner

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AOP-4241: Bullet Impact

- **Test Methods – *Modified***
  - Test Method 1: 3 12.7 mm AP M2 projectiles at 850 ± 20 m/s (600 ± 50 rounds/min)
  - Test Method 2: 1 12.7 mm AP M2 projectile at 850 ± 20 m/s
  - Test Method 3: 1 or several projectiles – projectile and velocity determined by THA

NEW

- Annex A: Recommendations aiming point and target area
- Annex B: Specifications 12.7 mm AP projectiles

NEW

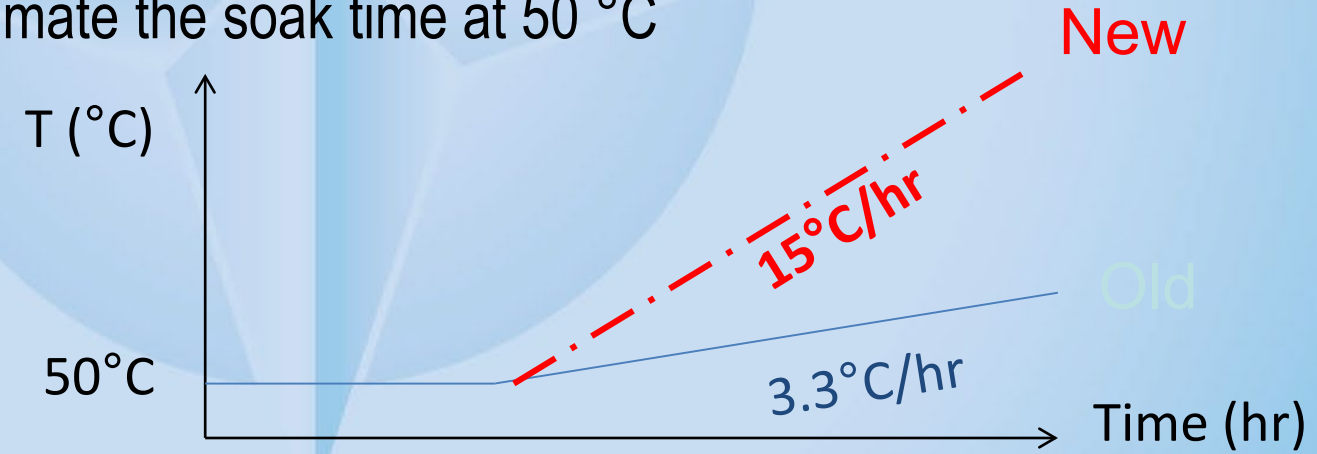


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AOP-4382: Slow Heating

- **Test Methods – *Modified***
  - Test Method 1: Preconditionning at 50 ± 3°C until thermal equilibrium of the test item, then Heating Rate **15 °C/hr** until reaction occurs
  - Test Method 2: Heating Rate determined by THA
  - Test Method 3 (UN HC): 3.3 °C/hr until reaction occurs – possibility to precondition at $T_{reaction - 55°C}$ (estimated)
- **Thermocouples – *Modified***
  - **6** required TC at 40-60 mm around the test item, rather than 4
- **Annex A:** Methods to estimate the soak time at 50 °C

NEW



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AOP-4396: Sympathetic Reaction

- **Single Test Method (based on donor initiation)**
  - If designed to detonate, initiate the donor munition in the design mode – ***Unchanged***
  - For munitions which are not designed to detonate, initiate the donor munition(s) with a credible threat that produces a worst-case response (e.g. shaped charge jet, explosive charge etc.) – ***Modified***
  - Sand / dirt etc. not to be used for simulating external confinement – ***Modified***
- **Baseline test:** recommendation to perform a baseline test in order to determine the blast, fragmentation and penetration signature of the donor test item(s), and the fragmentation and spatial distribution of inert acceptors.

NEW

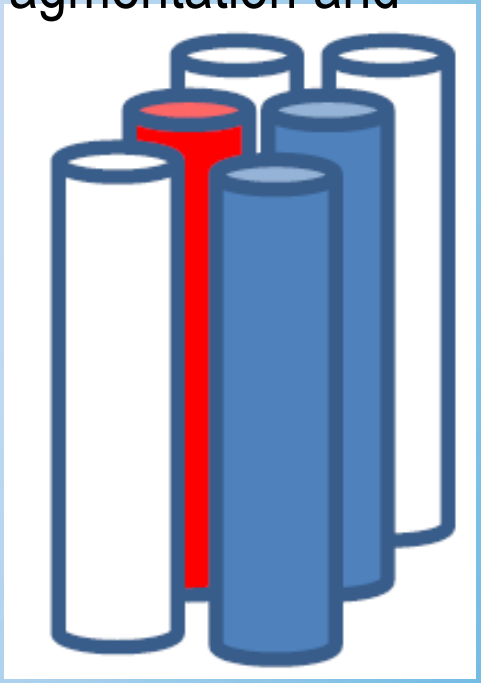
- **Additions in SRD AOP-39.1**
  - SR configuration examples
  - Clarification of what is the test item in SR tests

NEW

- **Addition of definitions for donor/acceptor munition in official NATO terminology**

NEW





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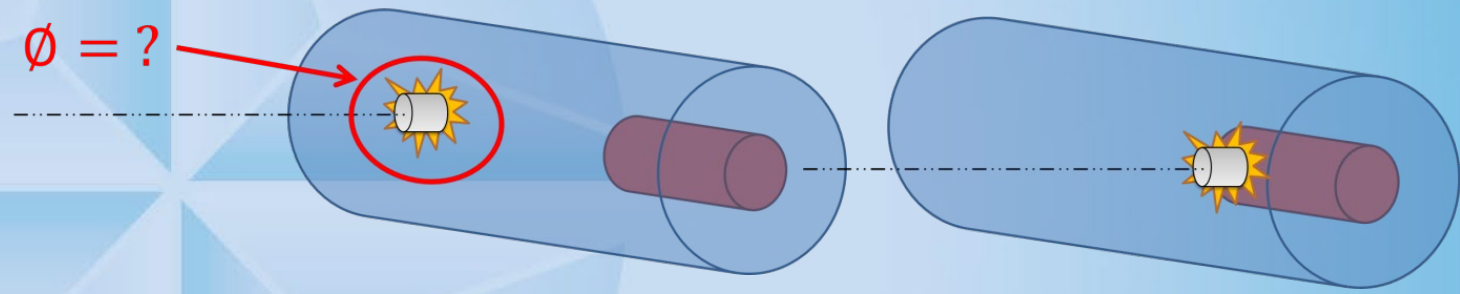
AOP-4496: Fragment Impact

- **Test Methods – *Unchanged***
  - Test Method 1: 2530 +/- 90 m/s
  - Test Method 2: 1830 +/- 60 m/s

NEW

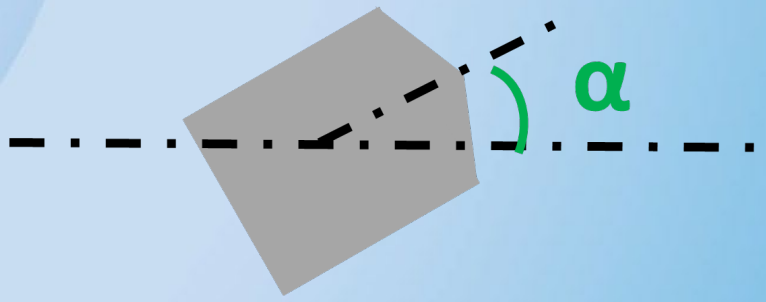
- **Accuracy at impact**
  - Set to ensure response mechanism under consideration is tested
  - Test item dependent
  - Approved by National Authorities

Small items: Hit the targeted area



NEW

- **Orientation of the fragment at impact**
  - Angular deviation **should** be limited to ± 10°



- **Brinell Hardess of the fragment – *Modified***
  - **190 < HB < 270**

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AOP-4526: Shaped Charge Jet

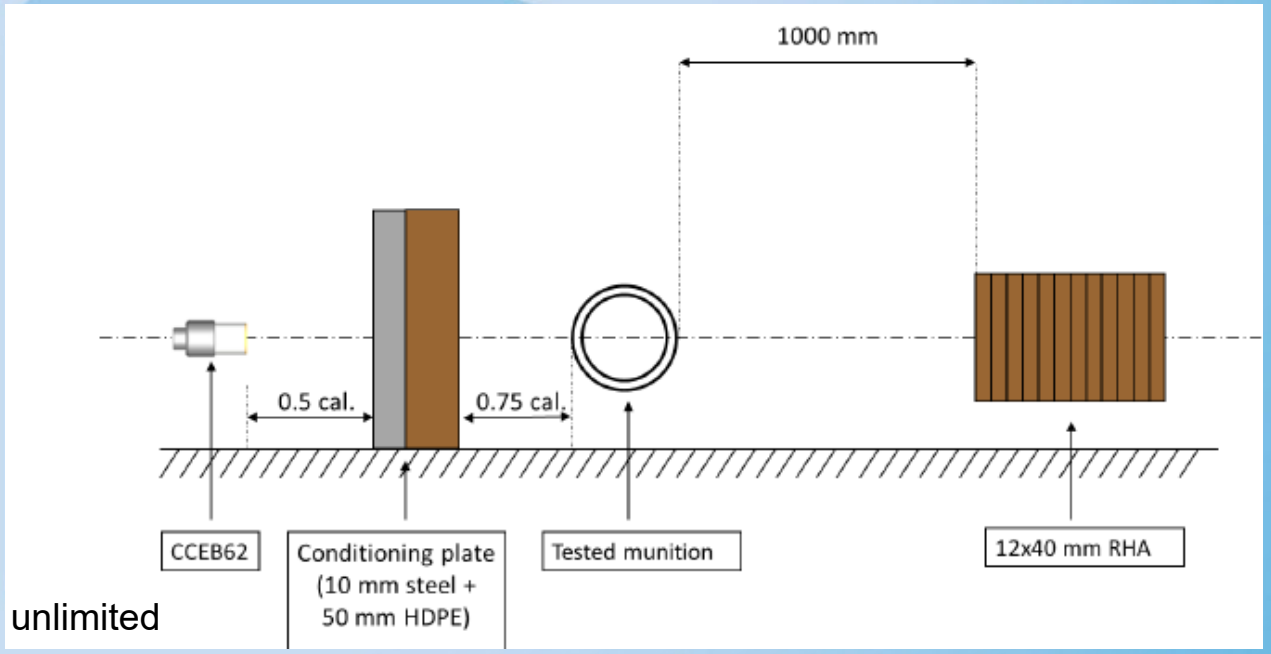
NEW

- **Test Methods – *Modified***
  - Test Method 1: SCJ characteristic of Rocket Propelled Grenade
  - Test Method 2: SCJ supported by means of a THA

NEW

- **Characterization of the SCJ**
  - V^2d at target between 120 and 140 $mm^3/\mu s^2$
  - Jet diameter at target between 2.5 and 3.5 mm

Previous: STANAG: Rockeye 50 mm
New: SC 81 mm LX-14 (USA); France : CCEB62



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IM Test AOP Version Update

- Due to long period of development, Editorial Working Group formed under AC/326 SG/B to review consistency & formatting of all 6 IM test AOPs
- Commenced Spring 2020 – virtual meetings every 2 weeks
- Work is subject of a separate presentation
  - Dan Pudlak – Working Group Chair

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Harmonization of IM & HC Standards

- Despite similarities in testing protocols employed by IM and Hazard Classification (HC) communities, a harmonized approach to testing has yet to be achieved
- IM/HC Harmonization Working Group established under AC/326 SG/B to explore the alignment of IM & HC standards and practices
- Work is subject of a separate presentation
  - Catherine Goodwin – Working Group Chair

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Concluding Remarks

- Updated editions of all 6 full-scale NATO IM test standards have now been promulgated, along with STANAG 4439 / AOP-39 / SRD AOP-39.1
  - Those involved in testing should ensure that they are now working to the latest standards.
- Ongoing work to review test standard consistency & formatting
  - Due to publish Version updates in 2021
- Longer-term work aimed at harmonizing HC and IM testing into a combined standard structure

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QUESTIONS?

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References

E. Baker and D. Hubble, “Slow Heating Test Thermal Equilibrium and Maximum Reaction Temperature”, 2019 Insensitive Munitions & Energetic Materials Technology Symposium, Seville, Spain, November 2019.

E. Baker, “An International Review of the Sympathetic Reaction Test”, MSIAC Report O-203, December 2018.

E. Baker, “Slow Heating Testing Survey and Historical Events Reviewt”, MSIAC Report O-195, October 2018.

D. Hubble, “Analysis of the Ramifications of Increasing the Slow Cook-off Test Heating Rate”, 2019 Insensitive Munitions & Energetic Materials Technology Symposium, Seville, Spain, November 2019.

C. Jacq, K. Tomasello, E. Baker and M. Sharp, “Review and Update of Insensitive Munitions Test Procedures”, 2019 Insensitive Munitions & Energetic Materials Technology Symposium, Seville, Spain, November 2019.

K. Tomasello, C. Jacq, E. Baker and M. Sharp, “NATO AC326 SG/B: Ammunition Systems Design and Assessment – IM Test STANAG updates”, 2019 Insensitive Munitions & Energetic Materials Technology Symposium, Seville, Spain, November 2019.

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Revisions and Improvements to the NATO Insensitive Munitions Test Doctrine Portfolio

Insensitive Munitions & Energetic Materials Technology Symposium (IMEMTS)

Virtual Teleconference

7-8 April 2021

Daniel J. Pudlak

DEPARTMENT OF THE ARMY
US ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
ARMAMENTS CENTER
PICATINNY ARSENAL, NEW JERSEY 07806-5000

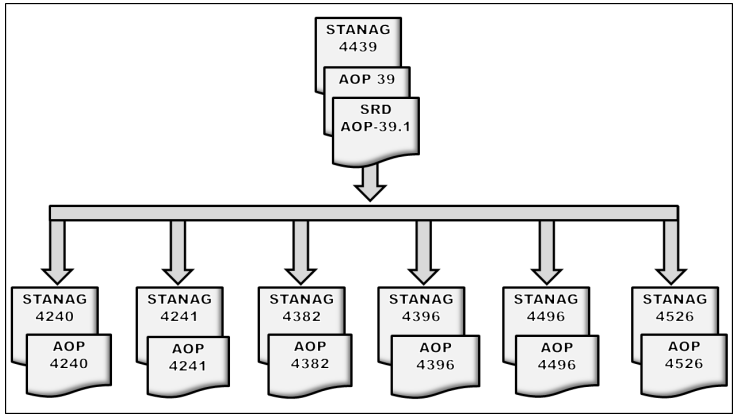
Phone: (973) 724-2223
Email: daniel.j.pudlak.civ@mail.mil

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BLUF

NATO AC/326 CASG SG/B - *IM Test AOP Standardization WG*

- North Atlantic Treaty Organization (NATO)
- Conference of National Armament Directors (CNAD) Ammunition Safety Group (AC/326) (CASG)
- Sub-Group B (SG/B) Ammunition Systems Design and Assessment
- Insensitive Munitions (IM) Test Allied Ordnance Publication (AOP) Standardization Working Group (WG)



K. Tomasello, 'NATO AC326 SG/B: Ammunition Systems Design and Assessment – IM Test STANAG updates', IMEMTS, Spain, 2019.

Objective: 'Clean up' NATO IM Doctrine Portfolio to ensure the text *effectively*, *accurately* and *consistently* supports the technical metrics.

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Introduction

NATO AC/326 CASG SG/B - *IM Test AOP Standardization WG*

- **Conceived** during Fall 2019 SG/B IM Sympathetic Reaction Test Custodial Working Group (SR-CWG) congregation (Koblenz, Germany).
- **Commissioned** as a result of action item to ensure consistency amongst the suite of the IM Portfolio documents (6 Test AOPs, 1 Policy AOP, 1 Guidance SRD) to ensure the new revisions comply with the WG decisions previously made.
- **Commenced** in Spring 2020 by reviewing the AOPs individually, and the respective Document Custodians were charged with incorporating the necessary changes.

    - **Members**:
        - NATO AC326 SG/B Chairman (Tomasello)
        - IM AOP Test WG Lead – (Pudlak)
        - Contractor Support – (Swierk)
        - MSIAC Support – (Ferran)
        - AOP Custodians
            - AOP-4240 – Netherlands (Bouma)
            - AOP-4241 – Germany (Hamhuis)
            - AOP-4382 – United States (Struck)
            - AOP-4396 – France (Jacq* / Weisse)
            - AOP-4496 – France (Jacq* / Weisse)
            - AOP-4526 – United States (Dr. Fuchs / Pudlak)
            - AOP-39 – France (Jacq* / Weisse)
            - AOP-39.1 – France (Jacq* / Weisse)
            - IM/HC – United Kingdom (Goodwin)

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Introduction

NATO CNAD AC/326 CASG SG/B - *IM Test AOP Standardization WG*

- Update to NATO IM Test Doctrine Portfolio:
  - ✓ Ensuring *consistency* amongst the test documents
  - ✓ During this process, IM Test AOP Standardization WG realized the AOPs could be improved:
    - *Textual changes* for clarity of intent
    - *Outline structural revision* to improve:
      - Readability
      - Flow logic
      - Consistency
    - *Match the latest NATO AOP Format* (provided by MSIAC)
  - ✓ Ultimately each of the updated 6 IM Test AOPs, 1 Policy AOP & 1 Guidance SRD:
    - Will undergo a *Version Update*
    - Will NOT undergo Ratification

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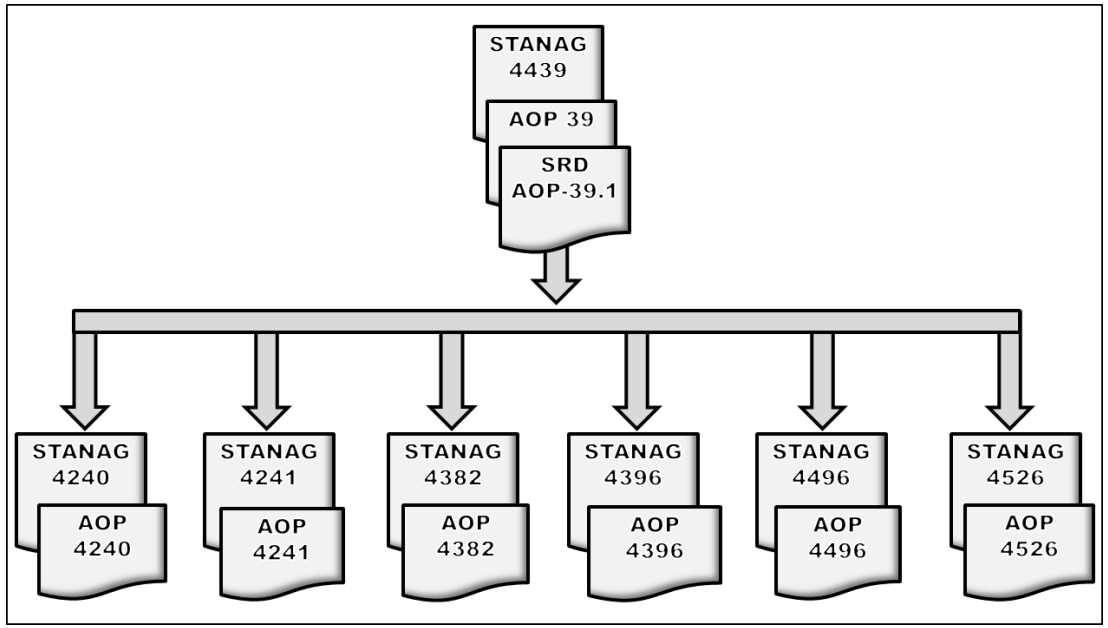
Purpose

- This presentation will ***briefly highlight*** the ***textual changes*** that were made, as well as the ***new format*** that was developed to synchronize these documents.

- Furthermore, this presentation will ***briefly discuss*** the ***subsequent changes*** that will be made to the ***related documents*** (e.g. AOP-39, SRD AOP-39.1, IM/HC AOPs, etc.) supporting these Test AOPs.

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Background – NATO Portfolio



K. Tomasello, 'NATO AC326 SG/B: Ammunition Systems Design and Assessment – IM Test STANAG updates', IMEMTS, Spain, 2019.

- New NATO Portfolio changes included *Re-organization* and *Re-structure*
  - **STANAGS** are now stand-alone Cover Documents
  - **Test AOPs** created to retain test requirements specific to each Test STANAG
  - **SRD** created to provide guidance and additional information (common amongst all Test AOPs)

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Background – Review Process

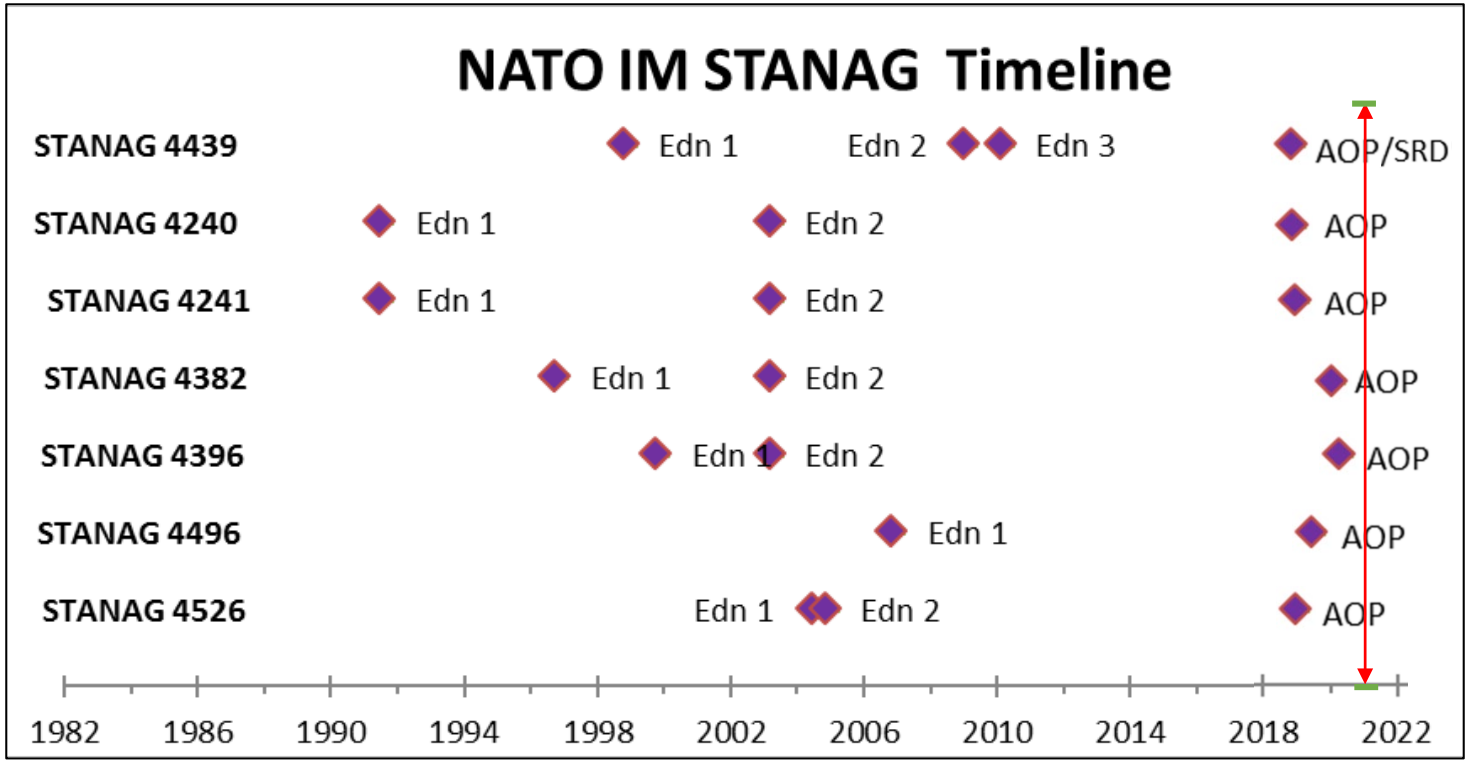
- Review and revision of the Test STANAGs started almost 10 years ago
  - During this process, new NATO changes called for formatting changes and creation of AOPs
- AOP-39 Review began during that timeframe
  - Became apparent this document needed to be stripped of unnecessary, outdated, irrelevant, inaccurate info
- Standards Related Document (SRD) was created as well
  - Obtained all the guidance and additional information from the STANAGs and AOP-39 that needed to be retained, but not lost
- Through-out this entire process, it was difficult to maintain consistency amongst the documents as well as retain justifications for changes made from one document to the next, for several reasons:
  - Participating members experience, background, and level participation
  - Language and interpretation
  - *Notes / records lost through-out the years

➢ Hence, the NATO IM Test AOP Standardization Working Group was created to ‘scrub’ the documents for clarity, readability, consistency, accuracy, and relevancy

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Background – Update Timeline

- ✓ STANAG / AOP 4240 (FH) : promulgated NOV 18
- ✓ STANAG / AOP 4241 (BI) : promulgated NOV 18
- ✓ STANAG / AOP 4496 (FI) : promulgated MAR 19
- ✓ STANAG / AOP 4526 (SCJI) : promulgated NOV 18
- ✓ STANAG / AOP 4382 (SH) : promulgated MAR 20
- ✓ STANAG / AOP 4396 (SR) : promulgated DEC 20



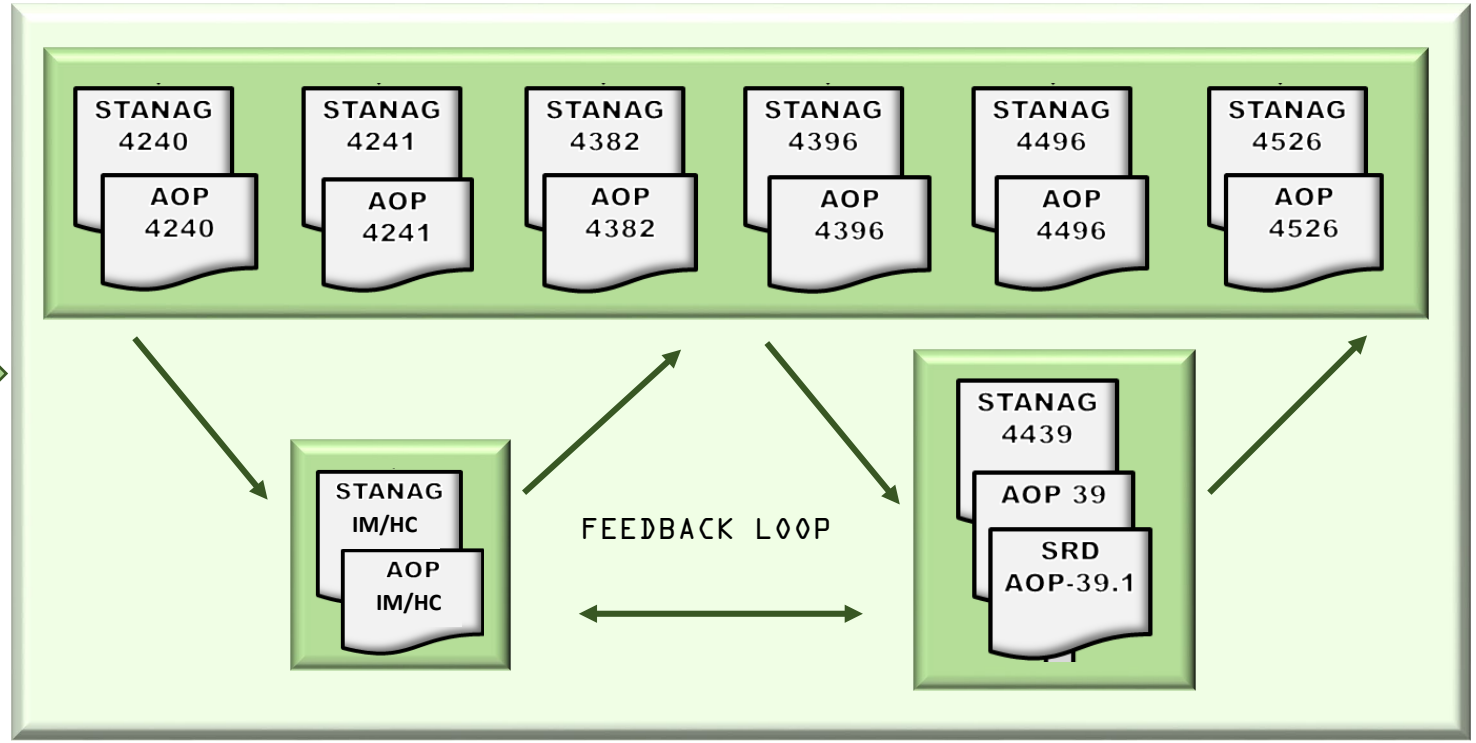
K. Tomasello, 'NATO AC326 SG/B: Ammunition Systems Design and Assessment – IM Test STANAG updates', IMEMTS, Spain, 2019.

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NATO IM Test Doctrine – Consistency Standardization / Synchronization Process

Version Updates (No changes to requirements)

- ✓ Revised individual Test AOPs
  - ✓ AOP-4396
  - ✓ AOP-4241
  - ✓ AOP-4496
  - ✓ AOP-4240
  - ✓ AOP-4382
  - ✓ AOP-4526
- ✓ Re-format all Test AOPs per NATO Template
- ✓ Compare all Test AOPs to ensure consistency
- ✓ Revise SRD based on changes to Test AOPs
- ✓ Revise AOP-39 based on changes to Test AOPs & SRD
- ➢ Final Review of all doc's for consistency
- ➢ Submit all Test AOPs, SRD & AOP-39 for Version Update



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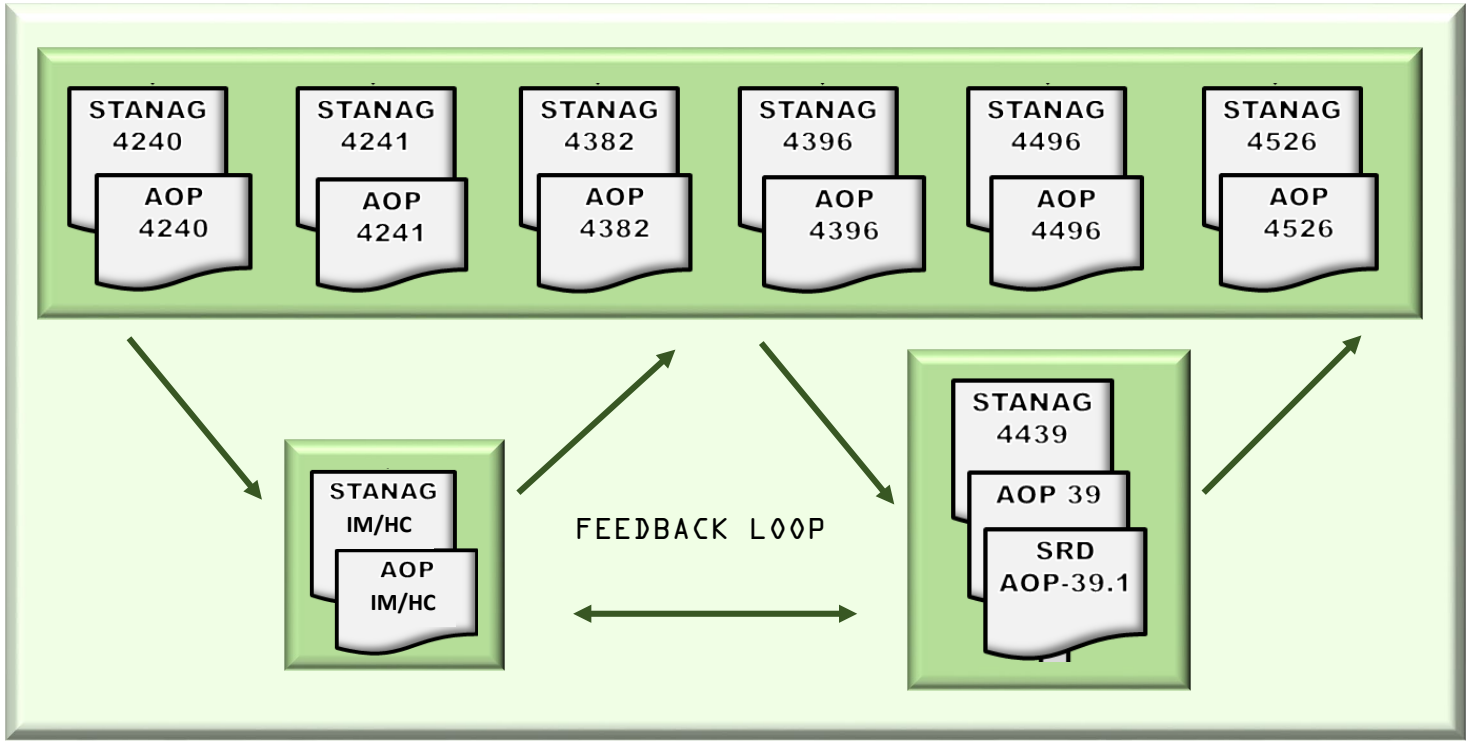
NATO IM Test Doctrine - *Revision Process Path Forward*

Version Updates (No changes to requirements) ← **Near Term**

- ✓ Revised individual Test AOPs (AOP-4396, AOP-4241, AOP-4496, AOP-4240, AOP-4382, AOP-4526)
- ✓ Re-format all Test AOPs per NATO Template
- ✓ Compare all Test AOPs to ensure consistency
- ✓ Revise SRD based on changes to Test AOPs
- ✓ Revise AOP-39 based on changes to Test AOPs & SRD
- ➢ Final Review of all doc's for consistency
- ➢ Submit all Test AOPs, SRD & AOP-39 for Version Update

Formal Updates ← **Looking Forward**

- ***Create IM/HC AOP*** based on changes to Test AOPs, SRD & AOP-39
- ***Revise Test AOPs based on technical proposals*** from IM Test AOP WG to AOP CWGs (AOP-4396, AOP-4241, AOP-4496, AOP-4240, AOP-4382, AOP-4526)
- ***Revise SRD*** based on changes to Test AOPs
- ***Revise AOP-39*** based on changes to Test AOPs & SRD
- Final Review of all doc's for consistency
- Submit Test AOPs, SRD & AOP-39 through Promulgation Process for ***Ratification***



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Progression & Results

- AOP-4396
  - Last AOP created during Winter (Jan-Mar) 2020
  - First AOP to get 'scrubbed' during Spring (Apr) 2020
    - Pro's
      - Benefitted from all the lessons learned through-out the AOP creation process
    - Con's
      - Guinea pig and benefited from none of the lessons learned through-out the AOP 'scrub' process.
  - Subsequently set the format for the new AOP Annex format
    - Historical Overview
    - Best Practices

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New Common AOP Annex Structure

- **Annexes**
  - Annex A – Best Practices
  - Annex B – Historical Overview
    - B.1 Revision Process
      - B.1.1 Creation of AOP-XXX
      - B.1.2 Changes from STANAG XXXX Ed.1
    - B.2 Background and Test Origins
      - B.2.1 Historical Overview

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AOP Review Process & Outcomes

In the following slides:

- AOP-4396 will be used as an example to demonstrate unique non-technical (no changes to requirements) considerations (Terminology, Definitions, Intentions) that were deliberated over, as well as unique improvements made since the initial AOPs were developed.

- AOP-4496 will be used as an example to demonstrate info moved from an AOP to the SRD.

- AOP-4526 will be used as an example to demonstrate the common non-technical (no changes to requirements) improvements made to the entire AOP body since the initial AOPs were developed.

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Deliberations & Unique Improvements to ***all*** AOPs

AOP-4396 will be used as an example to demonstrate unique **non-technical considerations (Terminology, Definitions, Intentions)** that were deliberated over, as well as **unique improvements** made since the initial AOPs were developed.

***Note** – For the purpose of this presentation, Words / Phrases are highlighted for the following purposes:*

- **Green – Indicates new / improved wording**
- **Orange – Indicates older wording that requires change / improvement, or belongs in SRD**
- **Red – Indicates older wording that was incorrect / misleading / obsolete**
- **Bold / Underlined annotate major topics for discussion**

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Terminology → Definitions → Intentions

❑ AOP-4396 Old Terms that needed Clear / Updated Definitions & New Terms

- Component
- Sub-component
- Component-level
- Design Mode
- Condition
- Packaged
- Unpackaged
- **Final Production Standard**
- Donor munition
- **Adjacent**
- **Unitization**
- **Palletization**
- National Authority

*Not all-inclusive; Also appear in other AOPs

❑ Differentiation of AOP-4396 Terms

- Test Article vs. Test Item vs. Item Under Test
- **Donor vs. Donor Test Item**
- **Acceptor vs. Acceptor Test Item**
- Test Item Configuration vs. Test Configurations
- Signature vs. Score vs. **Response vs. Reaction**
- Packaging vs. Packing
- Life Cycle Phase vs. Logistical Life Cycle
- Packaged vs. Unpackaged
- **Procedure vs. Method**
- All-Up-Round vs. Prototype
- Simulants vs. Inert Simulants vs. Mass Simulants
- **Energetic Component vs. Energetic Section**
- ***Energetic vs. *Explosive vs. Propellant vs. Pyrotechnic**

*Not all-inclusive; Also appear in other AOPs

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Terminology → **Definitions** → Intentions

- **Ex: AOP-4396, Section 1.5.3** The objective of the Sympathetic Reaction Test is to determine the response[1] of an acceptor test item(s)[3] when exposed to the worst-case credible reaction[4] of an identical donor test item. This test uses a worst-case credible configuration[5] experienced in its ~~l~~Life-~~c~~Cycle as defined in a THA. It is anticipated that the results of this test will be used to develop mitigation techniques to reduce the violence and consequences[6] of reactions[2] caused by accidents or hostile actions.

  - 1 - 'Response of an Acceptor Test Item' – use response when referring to / characterizing the munition's overall 'response' (i.e. score), or overall energetic event, for an entire test.
  - 2 - 'reactions' – when not necessarily referring to the overall response of an item, reaction may be used.
    - Ex: A primer *reacted* at timestamp x during the cook-off.
  - 3 - 'Response of an Acceptor Test Item' – *'Acceptor'* or *'Test Item'* not technically specific enough.
    - *'Acceptor'* may not refer to the entire Acceptor Test Item, for example, it may refer to one round in a box of multiple rounds.
    - *'Test Item'* – encompasses both the Acceptor Test Item and the Donor Test Item.
  - 4 - 'Worst-case Credible Reaction' – implies the worst credible reaction, that the donor test item can produce, must be exposed to the acceptor.
  - 5 - 'Worst-case Credible Configuration' – implies the donor test item configuration that produces the worse credible reaction must be utilized for the test.
  - 6 - 'consequences' – certain participating member nations have particular focuses / interests and we decided as a group to include their inputs/request/proposals if they do not deter the test from meeting the requirements.

Note – These are not the official NATO terms for these words / phrases. They are simply paraphrased for this presentation.

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Terminology → Definitions → **Intentions**

❖ **Ex: AOP-4396, Section 2.2.2 Test Requirements (*Initial AOP, from STANAG*)**

d. External Confinement. Any confinement should represent that of a typical storage confinement. Confinement may be simulated with sandbags or sand/earth-filled containers stacked around the test stack at least 1m thick in all directions. If an existing external confinement is likely to alter the test result, the confinement should be simulated in the test. Both partial and complete confinement are possible, e.g., walls in two dimensions or an enclosed magazine. The latter can be simulated by a test structure for which the ratio of the volume of active and inert munitions to the volume of the test cell approximates that existing in the actual operational or storage configuration. Variations of up to 20% are acceptable. It is not appropriate to use inert items in the test arrangement simulating the unconfined situation, because they contribute to confinement without the possibility of reacting.

❑ **"External confinement should represent typical storage confinement"**

- ➢ Most of the content in the IM STANAGs were taken directly from Hazard Classification (HC) documents (e.g. Orange Book)
  - For example: 'walls of ma… d 'test structures'
    - Indicate wording f… IM documents
    - Orange Book lists several examples of loading docks, bays, concrete walls, etc. to replace
- ➢ Historically, HC addressed 'storage configurations' and IM addressed 'operational configurations' like IM prefers
  - Without deliberating, they seem one in the same, however they are not necessarily.
- ➢ In effort to address IM concerns, we focus configurations more on the munition's logistical / operational configurations per their THA.
- ➢ For SR, historically, IM focuses on munition response from Un-Confined Test
- ➢ Confined test has been redefined / clarified in previous revision (Annex B)
  - UN expressed interest in conforming based on recent dimensional analysis conducted on NATO pallets and stack sizes.

BLUF:

- **IM doc's taken from HC**
- **Re-written to address IM concerns**
  - **Incorporated IM/HC Harmonization**
- **Include Technical Justifications for New/Legacy Metrics**

❖ **Ex: AOP-4396, Section 2.2.2 Test Requirements (*Revised AOP*)**

d. External Confinement: If an existing external confinement is likely to alter the test result, the confinement should be simulated in at least one of the two minimum required tests. Any confinement should represent that of a typical storage/transport confinement. Based on the information provided in Annex A, confinement thickness will depend on the lifecycle situation the specific test is supposed to simulate (guidance of at least 1 meter deep in all directions around the test item), and is typically represented in the palletized configuration, based on the logistical lifecycle cited in the THA. The confinement should include the packaging, unitization, and palletization material as per the packaging, unitization and palletization drawings. Common materials as per these drawings should be used to accurately represent the confinement of the fielded munition, however inert munitions and mass simulants may be used if a technical justification indicates the same conclusions will result and is approved by the National Authority. **Safety concerns shall prohibit the use of sand, dirt or similar loose granular material for simulating external confinement.** Both partial and complete confinement test configurations are possible.

❑ **'guidance of at least 1 meter deep in all directions around the test item…and is typically represented in the palletized configuration, based on the logistical lifecycle cited in the THA'**

- ➢ See Annex B for dimensional analysis of NATO pallets and stack sizes and the hypothesized technical reasoning for this metric.
- ➢ Accepted by all member nations
- ➢ Added THA as it was not emphasized well previously

❑ **Materials should be used per the packaging, unitization, palletization, etc. drawings**

❑ **Sand bags no longer tolerable for several reasons:**

- ➢ Sand is an excellent fragment, shock wave and fire suppressor
  - It inhibits the true response of the Acceptor Test Item
- ➢ Unknown / unreacted live energetics get buried in sand
  - Unsafe for test facilities
  - Difficult to find / evaluate test results

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Common Improvements to ***all*** AOPs

AOP-4496 will be used as an example to demonstrate items that were moved from an **AOP to the SRD**.

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Info from AOP → SRD

From 2.2. Test Types

4. Aim points selection: Aim point shall be selected to create the most stressing condition on the target energetic. The aim point shall also represent a credible exposure condition, based on the THA. The shotline should avoid impact locations of low probability that may lead to irreproducible reactions. This includes, but is not limited to, welds seams or joints. The first test shall be conducted with an aim point and shotline at the centre of the energetic component. The second test shall be conducted on the most vulnerable area. Guidance for choosing the aim point and shotline can be found in SRD AOP-39.1. The aim point and shotline for each test should be approved by the national authority prior to testing.

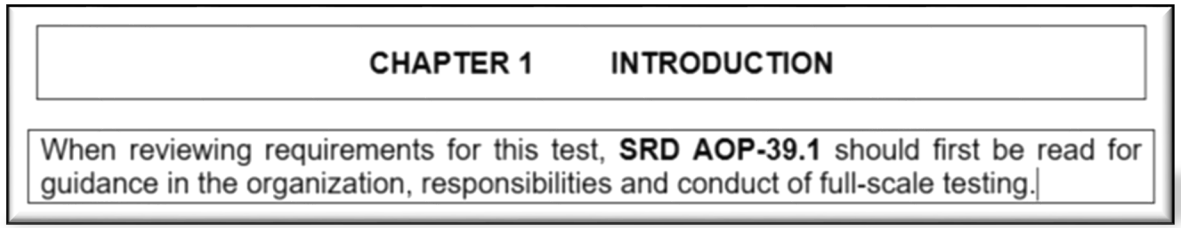
WEISSE Quentin IETA
To move in the SRD.
There is no mention of the THA in the part B.3.3 (Aim/Impact point – shotline) of the SRD.

7. Methods shall be established to assure the fragment is aimed at the selected aimpoint and that it follows the desired path through the munition. The likelihood of getting a violent response, will normally be maximized by choosing a shotline, which provides the longest possible path length through the energetic material. However, unlikely shotlines should be avoided, which are aimed at components that are quite small when compared to the bulk of the explosive, propellant or aimed at unlikely angles. Prior to testing, shotlines should be agreed to by the national authority. In this regard, the following considerations may apply: if the energetic material contains a cavity of significant size (such as the bore of a rocket motor), aim the fragment to pass perpendicularly through the cavity. (It has been observed that such cavities can promote the occurrence of violent reactions).

WEISSE Quentin IETA
To move in the SRD such as for AOP-4241

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CHAPTER 1 INTRODUCTION

When reviewing requirements for this test, **SRD AOP-39.1** should first be read for guidance in the organization, responsibilities and conduct of full-scale testing.

Due to the portfolio structure, improvements were made to maximize clarification as much as possible.

- For example:
  - Requirements were moved from STANAG → AOP
    - AOP should no longer contain guidance
  - It is apparent (from surveys executed during the Custodial Working Group meetings), that even the WG members have different perceptions of many of the topics:
    - Terms
    - Definitions
    - Methods / Procedures
    - Quantities
    - Qualities
    - Etc.
  - SRD contains guidance and additional / supporting information
    - ✓ Added the above note to the top of the first page in Chapter 1

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Common Improvements to ***all*** AOPs

AOP-4526 *will be used as an example* to demonstrate the common **non-technical improvements to each section of the AOP body** since the initial AOPs were developed.

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New Common AOP Body Structure

- Cover Page
- NSO NATO Letter of Promulgation
- Record of Observations
- Record of Specific Reservations
- Table of Contents
- Chapter 1
  - 1.1 Annexes
  - 1.2 Related Documents
  - 1.3 AIM
  - 1.4 Agreement
  - 1.5 Definitions
  - 1.6 General
  - 1.7 Test Limitations
- Chapter 2
  - 2.1 Test Item Configuration
  - 2.2 Test Details
    - 2.2.1 Test Methods
    - 2.2.2 Test Requirements
      - 2.2.2.1 Generic
      - 2.2.2.2 Specific
    - 2.2.3 Test Set-Up
    - 2.2.4 Number of Tests
  - 2.3 Documentation and Compliance
  - 2.4 Observations and Records
  - 2.5 Evaluation of Test Results
- Annex A
- Annex B

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Common Improvements to ***all*** AOPs

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❖Annexes

1.1. ANNEXES

A. Shaped Charge 81 mm, LX-14 (USA)
B. Shaped Charge CCEB 62 (French)
C. Historical Overview

→

1.1 ANNEXES

A. Best Practices
B. Historical Overview

ANNEX A BEST PRACTICES....A-1
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B.2 CHANGES FROM STANAG 4526 EDITION 2....B-1

Restructured format and reorganized information / sections in a more categoric manner.

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❖ 1.2 RELATED DOCUMENTS

1.2 RELATED DOCUMENTS		
STANAG 4439	Policy for Introduction and Assessment of Insensitive Munitions (IM)	
AOP-39	Policy for Introduction and Assessment of Insensitive Munitions (IM)	
SRD AOP-39.1	Guidance on the Organization, Conduct and Reporting of Full-scale Tests	
STANAG 4526	Shaped Charge Jet Impact Test Procedures for Munitions	
AASTP-03	Manual of NATO Safety Principles for the Hazard Classification of Military Ammunition and Explosives	Suggest removal as SCJ not used in HC testing
United Nations	Manual of Tests and Criteria (ST/SG/AC.10/11)	Suggest removal as SCJ not used in HC testing

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❖ 1.~~2~~3 AIM

The aim of this Standard[1] is to specify the test requirements and procedures to provide evidence of the reaction[2] of munitions and weapon systems to the threats represented from[3] being struck[4] by a shaped charge weapon[5].	The aim of this AOP[1] is to specify the test requirements and procedures to provide evidence of the response[2] of munitions and weapon systems to the threats represented by[3] being impacted[4] by small arms projectile(s) [5].

- ➢ Standard[1] – Previous document
- ✓ AOP[1] – New document
- ➢ Reaction[2] – Constituent / Included in the description / definition of a m*unition response*
- ✓ Response[2] – Describes the wholesome energetic event; Includes single / multiple r*eactions*
- ➢ from[3] – Bad grammar
- ✓ by[3] – Better grammar
- ➢ struck[4] – Not technically accurate
- ✓ impacted[4] – Technically accurate
- ➢ shaped charge weapon[5] – Specific to individual AOP
- ✓ small arms projectile(s) [5] – Specific to individual AOP

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❖1.~~3~~4 AGREEMENT

- Participating nations agree that the requirements and ~~procedures~~ methods incorporated in this AOP will be used for determining the response of munitions and weapon systems to a shaped charge jet impact represented by the most prevalent threat (currently the RPG) or more appropriate threat.

- Participating nations further agree that national standards, orders, manuals and instructions implementing this AOP will include a reference to the STANAG 4526 for purposes of identification.

- No departure may be made from this agreement without consultation with the NATO Tasking Authority~~ies/Delegated Tasking Authorities (TA/DTAs)~~. Nations may propose changes at any time to the ~~TA/DTAs~~ NATO Tasking Authority where they will be processed in the same manner as the original agreement.

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❖1.45 DEFINITIONS

- For the purpose of this document, definitions of terms to be used to describe test details and events are given in the NATO Terminology ~~Management System~~ Database (NATOTerm) that is available by reference for all Allied Publications[1].

- [1]https://nso.nato.int/natoterm/

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❖1.5 GENERAL

1. Minimizing the reaction of ordnance to shaped charge jet impact is an ongoing commitment of weapons designers to ensure that the safety of personnel and materiel will not be unduly jeopardized.
2. This Standard addresses the situation where munitions and weapon systems may be struck by a shaped charge projectile most probably whilst on operations. This can have a significant consequence for personnel and equipment from the response of their own munitions and weapon systems to such a threat.
3. Other tests may be required to evaluate the response of munitions in tactical situations, such as when stowed on armored vehicles.

→

❖1.~~5~~6 GENERAL

1. Efforts to minimize the violence of the reaction of munitions impacted by a shaped charge jet is a continuing commitment of weapons designers in order that the safety of personnel and materiel will not be unduly jeopardized.
2. This AOP addresses the situation where munitions and weapon systems are impacted by a shaped charge jet. This can occur in peacetime as the result of an accident, a dissident/saboteur activity, or an operation as a consequence of enemy action, which can result in a significant compromise of safety.
3. The objective of the Shaped Charge Jet Impact test is to determine the response of the munition(s) when subjected to a defined shaped charge jet.

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added

deleted

sent to SRD

2.2. TEST TYPES

There are two methods for performing the Shaped Charge Jet Impact Test; a "Standard Test" and an "Alternative Test" for Munitions. For the selected test, subject the munition in either a logistical and/or tactical configuration, to a jet from a shaped charge, as documented in an approved ŧTest ~~p~~Plan.

a. Method 1 (Standard ~~Threat Test~~): Using the general guidance specified herein, subject the ~~t~~Test ~~i~~Item to the jet from a shaped charge representing the rocket propelled grenades, as described in Section 2.~~5~~2.~~1~~2, ~~m~~Method 1, of this document. The complete characterization of the jet used for the test shall be conducted to meet the requirements of this AOP, and shall be provided or referenced. Examples of tests fulfilling these requirements can be found in the ~~a~~Annexe~~s~~ A.

b. Method 2 (Alternative ~~Threat Test~~): Using the general guidance specified herein, subject a ~~t~~Test ~~i~~Item to a well characterized shaped charge jet as documented in a Threat Hazard Assessment (THA). The jet should be fully characterized and reported, as documented in ~~s~~Section~~s~~ 2.~~5~~2.~~1~~2~~, method 2~~.

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2.4 SHAPED CHARGE

1.

2.

2.5 JET CHARACTERISTIC REQUIREMENTS

2.5.1 Characterization of the Shaped Charge Jet

1.

2.

a….

…f.

3.

a…

…f.

2.5.2 Measurements

1.

2.

2.5.3 Shot line



2.2.2 Test Requirements

2.2.1 Generic

1. Shaped Charge Jet Requirements

a.

b.

2. Jet Characteristic Requirements

a…

…d

2.2.2.2 Specific

1.

a…

…f

2.

a…

…f

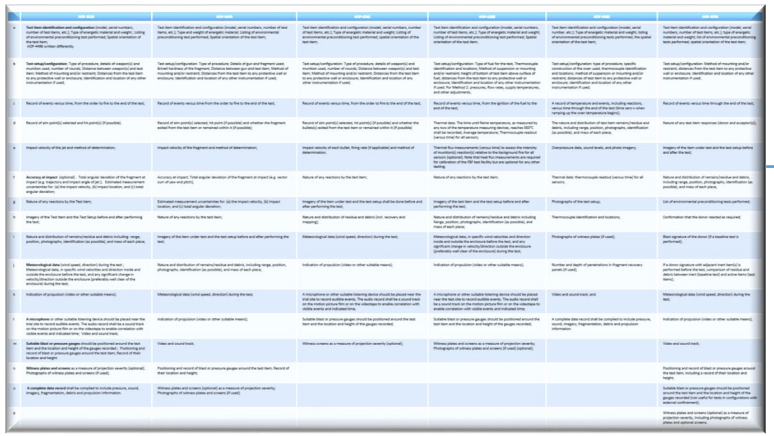
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	EX: AOP-4526 OBSERVATIONS & RECORDS
a	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight ; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;
b	Test setup/configuration: Type of procedure; Details of shaped charge used; Distance between weapon(s) and test item; Method of mounting and/or restraint; Distances between the shaped charge, the conditioning plate, and the test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;
c	Record of events versus time, from the order to fire to the end of the test;
d	Record of aim point(s) selected;
e	Details of shaped charge jet characteristics and conditioning plate.
f	No other specific observation/measurement found;
g	Nature of any reactions by the Test Item;
h	Photo Imagery of the Test Item and the Test Setup before and after performing the test;
i	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;
j	Meteorological data (wind speed, direction) during the test;
k	Indication of propulsion (video or other suitable means);
l	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time;
m	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height;
n	Witness plates and screens as a measure of projection severity (optional); Photographs of witness plates and screens (if used); Number and depth of penetrations in fragment recovery panels (if used);
o	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information;

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OBSERVATIONS & RECORDS



Demonstration of Revision Process





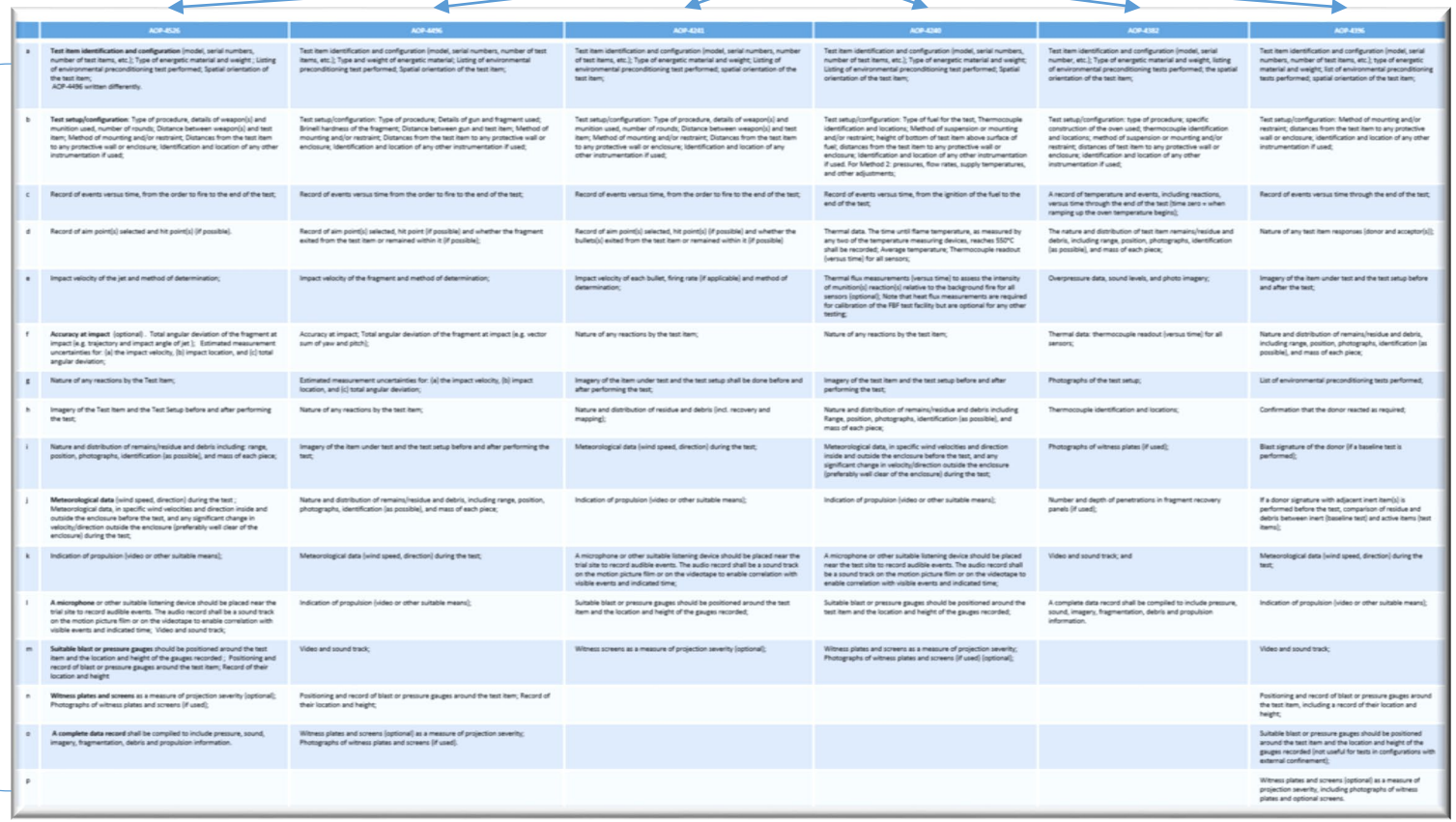
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OBSERVATIONS & RECORDS

- ***Created*** **Observations & Records Comparison Chart**
  - Cut & pasted directly from the current AOPs
    - Content originally from STANAGs
      - No particular order
      - Duplications
      - Inconsistent
      - Missing / Addn'l req's
      - Misleading wording
      - Inaccurate terms

AOPs

Ob's & Rec's



	AOP-4526	AOP-4496	AOP-4241	AOP-4240	AOP-4382	AOP-4396
a	**Test item identification and configuration** (model, serial numbers, number of test items, etc.); Type of energetic material and weight ; Listing of environmental preconditioning test performed; Spatial orientation of the test item; AOP-4496 written differently.	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type and weight of energetic material; Listing of environmental preconditioning test performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning test performed; spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning test performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial number, etc.); Type of energetic material and weight, listing of environmental preconditioning tests performed; the spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); type of energetic material and weight; list of environmental preconditioning tests performed; spatial orientation of the test item;
b	**Test setup/configuration**: Type of procedure, details of weapon(s) and munition used, number of rounds; Distance between weapon(s) and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of procedure; Details of gun and fragment used; Brinell hardness of the fragment; Distance between gun and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of procedure, details of weapon(s) and munition used, number of rounds; Distance between weapon(s) and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of fuel for the test; Thermocouple identification and locations; Method of suspension or mounting and/or restraint; height of bottom of test item above surface of fuel; distances from the test item to any protective wall or enclosure; identification and location of any other instrumentation if used. For Method 2: pressures, flow rates, supply temperatures, and other adjustments;	Test setup/configuration: type of procedure; specific construction of the oven used; thermocouple identification and locations; method of suspension or mounting and/or restraint; distances of test item to any protective wall or enclosure; identification and location of any other instrumentation if used;	Test setup/configuration: Method of mounting and/or restraint; distances from the test item to any protective wall or enclosure; identification and location of any other instrumentation if used;
c	Record of events versus time, from the order to fire to the end of the test;	Record of events versus time from the order to fire to the end of the test;	Record of events versus time, from the order to fire to the end of the test;	Record of events versus time, from the ignition of the fuel to the end of the test;	A record of temperature and events, including reactions, versus time through the end of the test (time zero = when ramping up the oven temperature begins);	Record of events versus time through the end of the test;
d	Record of aim point(s) selected and hit point(s) (if possible).	Record of aim point(s) selected, hit point (if possible) and whether the fragment exited from the test item or remained within it (if possible);	Record of aim point(s) selected, hit point(s) (if possible) and whether the bullet(s) exited from the test item or remained within it (if possible)	Thermal data. The time until flame temperature, as measured by any two of the temperature measuring devices, reaches 550°C shall be recorded; Average temperature; Thermocouple readout (versus time) for all sensors;	The nature and distribution of test item remains/residue and debris, including range, position, photographs, identification (as possible), and mass of each piece;	Nature of any test item responses (donor and acceptor(s));
e	Impact velocity of the jet and method of determination;	Impact velocity of the fragment and method of determination;	Impact velocity of each bullet, firing rate (if applicable) and method of determination;	Thermal flux measurements (versus time) to assess the intensity of munition(s) reaction(s) relative to the background fire for all sensors (optional); Note that heat flux measurements are required for calibration of the FBF test facility but are optional for any other testing;	Overpressure data, sound levels, and photo imagery;	Imagery of the item under test and the test setup before and after the test;
f	**Accuracy at impact** (optional) . Total angular deviation of the fragment at impact (e.g. trajectory and impact angle of jet); Estimated measurement uncertainties for: (a) the impact velocity, (b) impact location, and (c) total angular deviation;	Accuracy at impact; Total angular deviation of the fragment at impact (e.g. vector sum of yaw and pitch);	Nature of any reactions by the test item;	Nature of any reactions by the test item;	Thermal data: thermocouple readout (versus time) for all sensors;	Nature and distribution of remains/residue and debris, including range, position, photographs, identification (as possible), and mass of each piece;
g	Nature of any reactions by the Test Item;	Estimated measurement uncertainties for: (a) the impact velocity, (b) impact location, and (c) total angular deviation;	Imagery of the item under test and the test setup shall be done before and after performing the test;	Imagery of the test item and the test setup before and after performing the test;	Photographs of the test setup;	List of environmental preconditioning tests performed;
h	Imagery of the Test Item and the Test Setup before and after performing the test;	Nature of any reactions by the test item;	Nature and distribution of residue and debris (incl. recovery and mapping);	Nature and distribution of remains/residue and debris including Range, position, photographs, identification (as possible), and mass of each piece;	Thermocouple identification and locations;	Confirmation that the donor reacted as required;
i	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;	Imagery of the item under test and the test setup before and after performing the test;	Meteorological data (wind speed, direction) during the test;	Meteorological data, in specific wind velocities and direction inside and outside the enclosure before the test, and any significant change in velocity/direction outside the enclosure (preferably well clear of the enclosure) during the test;	Photographs of witness plates (if used);	Blast signature of the donor (if a baseline test is performed);
j	**Meteorological data** (wind speed, direction) during the test ; Meteorological data, in specific wind velocities and direction inside and outside the enclosure before the test, and any significant change in velocity/direction outside the enclosure (preferably well clear of the enclosure) during the test;	Nature and distribution of remains/residue and debris, including range, position, photographs, identification (as possible), and mass of each piece;	Indication of propulsion (video or other suitable means);	Indication of propulsion (video or other suitable means);	Number and depth of penetrations in fragment recovery panels (if used);	If a donor signature with adjacent inert item(s) is performed before the test, comparison of residue and debris between inert (baseline test) and active items (test items);
k	Indication of propulsion (video or other suitable means);	Meteorological data (wind speed, direction) during the test;	A microphone or other suitable listening device should be placed near the trial site to record audible events. The audio record shall be a sound track on the motion picture film or on the videotape to enable correlation with visible events and indicated time;	A microphone or other suitable listening device should be placed near the test site to record audible events. The audio record shall be a sound track on the motion picture film or on the videotape to enable correlation with visible events and indicated time;	Video and sound track; and	Meteorological data (wind speed, direction) during the test;
l	**A microphone** or other suitable listening device should be placed near the trial site to record audible events. The audio record shall be a sound track on the motion picture film or on the videotape to enable correlation with visible events and indicated time; Video and sound track;	Indication of propulsion (video or other suitable means);	Suitable blast or pressure gauges should be positioned around the test item and the location and height of the gauges recorded;	Suitable blast or pressure gauges should be positioned around the test item and the location and height of the gauges recorded;	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	Indication of propulsion (video or other suitable means);
m	**Suitable blast or pressure gauges** should be positioned around the test item and the location and height of the gauges recorded ; Positioning and record of blast or pressure gauges around the test item; Record of their location and height	Video and sound track;	Witness screens as a measure of projection severity (optional);	Witness plates and screens as a measure of projection severity; Photographs of witness plates and screens (if used) (optional);		Video and sound track;
n	**Witness plates and screens** as a measure of projection severity (optional); Photographs of witness plates and screens (if used);	Positioning and record of blast or pressure gauges around the test item; Record of their location and height;				Positioning and record of blast or pressure gauges around the test item, including a record of their location and height;
o	**A complete data record** shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).				Suitable blast or pressure gauges should be positioned around the test item and the location and height of the gauges recorded (not useful for tests in configurations with external confinement);
p						Witness plates and screens (optional) as a measure of projection severity, including photographs of witness plates and optional screens.

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OBSERVATIONS & RECORDS

- ***Utilized* Observations & Records Comparison Chart**
  - Evaluated Ob's & Rec's for each individual AOP
  - Compared differences and similarities
  - Created an organized matrix (a-p)
  - Added / Omitted where appropriate
  - Re-worded for accuracy and clarity
  - Ensured terms match AOP content
  - Retained requirements
  - Omitted guidance

AOPs

Ob's & Rec's



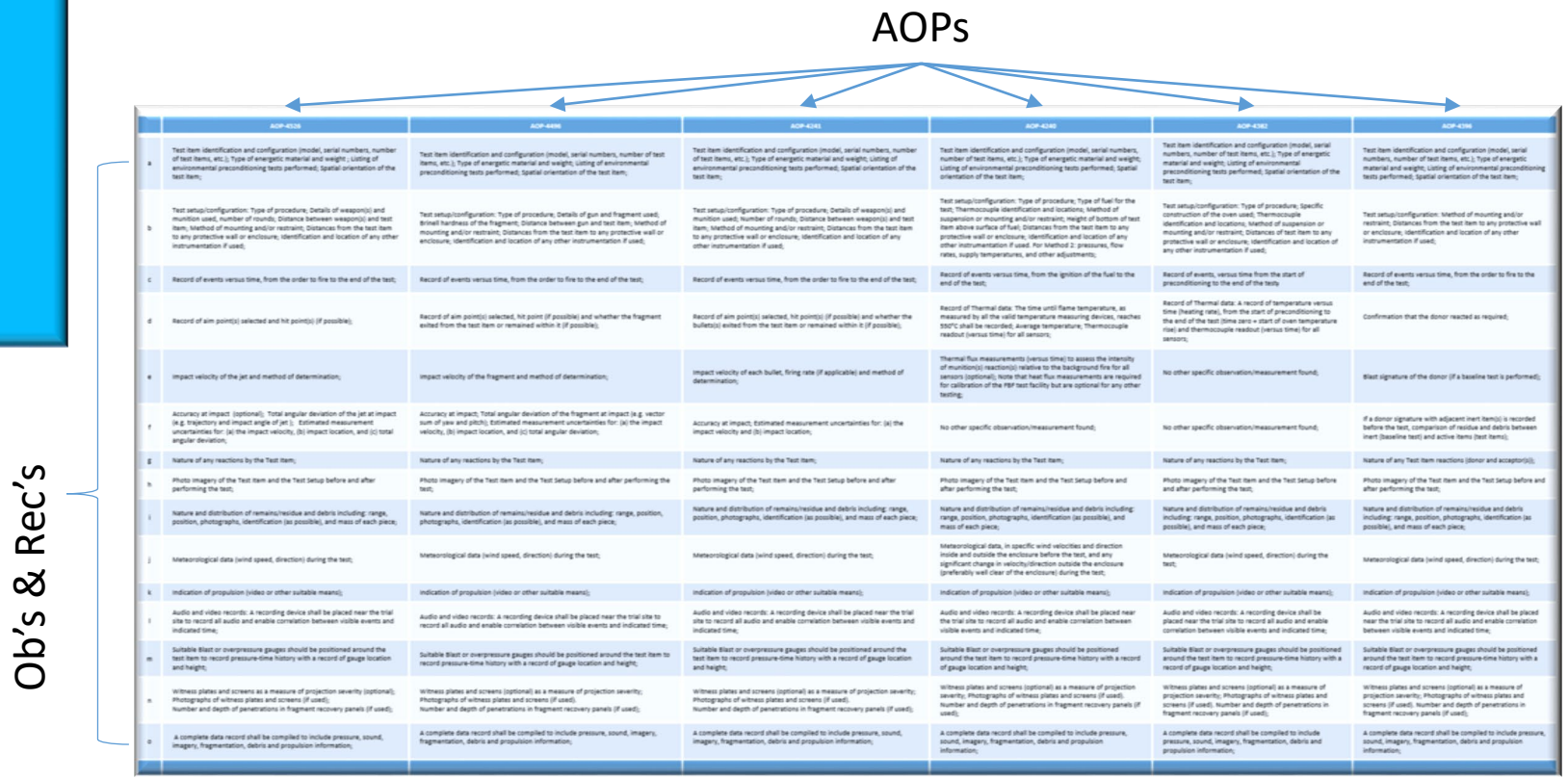
	AOP-4526	AOP-4496	AOP-4241	AOP-4240	AOP-4382	AOP-4196
a	**Test item identification and configuration** (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;	Test item identification and configuration (model, serial numbers, number of test items, etc.); Type of energetic material and weight; Listing of environmental preconditioning tests performed; Spatial orientation of the test item;
b	**Test setup/configuration**: Type of procedure; Details of weapon(s) and munition used, number of rounds; Distance between weapon(s) and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of procedure; Details of gun and fragment used; Brinell hardness of the fragment; Distance between gun and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of procedure; Details of weapon(s) and munition used; Number of rounds; Distance between weapon(s) and test item; Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Type of procedure; Type of fuel for the test; Thermocouple identification and locations; Method of suspension or mounting and/or restraint; Height of bottom of test item above surface of fuel; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used. For Method 2: pressures, flow rates, supply temperatures, and other adjustments;	Test setup/configuration: Type of procedure; Specific construction of the over used; Thermocouple identification and locations; Method of suspension or mounting and/or restraint; Distances of test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;	Test setup/configuration: Method of mounting and/or restraint; Distances from the test item to any protective wall or enclosure; Identification and location of any other instrumentation if used;
c	Record of events versus time, from the order to fire to the end of the test;	Record of events versus time, from the order to fire to the end of the test;	Record of events versus time, from the order to fire to the end of the test;	Record of events versus time, from the ignition of the fuel to the end of the test;	Record of temperature and events, including reactions, versus time through the end of the test (time zero = when ramping up the oven temperature begins); Thermal data: A record of temperature versus time and time of events, including reactions, through the end of the test (start of oven temperature rise = t0) and thermocouple readout (versus time) for all sensors;	Record of events versus time, from the order to fire to the end of the test;
d	Record of aim point(s) selected and hit point(s) (if possible).	Record of aim point(s) selected, hit point (if possible) and whether the fragment exited from the test item or remained within it (if possible);	Record of aim point(s) selected, hit point(s) (if possible) and whether the bullet(s) exited from the test item or remained within it (if possible);	Record of Thermal data: The time until flame temperature, as measured by all the valid temperature measuring devices, reaches 550°C shall be recorded; Average temperature; Thermocouple readout (versus time) for all sensors;	Record of Thermal data: Heating rate, Thermocouple readout (versus time) for all sensors;	Confirmation that the donor reacted as required;
e	Impact velocity of the jet and method of determination;	Impact velocity of the fragment and method of determination;	Impact velocity of each bullet, firing rate (if applicable) and method of determination;	Thermal flux measurements (versus time) to assess the intensity of munition(s) reaction(s) relative to the background fire for all sensors (optional); Note that heat flux measurements are required for calibration of the FBF test facility but are optional for any other testing;	No other specific observation/measurement found	Blast signature of the donor (if a baseline test is performed);
f	**Accuracy at impact** (optional): Total angular deviation of the jet at impact (e.g. trajectory and impact angle of jet.); Estimated measurement uncertainties for: (a) the impact velocity, (b) impact location, and (c) total angular deviation;	Accuracy at impact; Total angular deviation of the fragment at impact (e.g. vector sum of yaw and pitch); Estimated measurement uncertainties for: (a) the impact velocity, (b) impact location, and (c) total angular deviation;	Accuracy at impact; Estimated measurement uncertainties for: (a) the impact velocity and (b) impact location;	No other specific observation/measurement found	No other specific observation/measurement found	If a donor signature with adjacent inert item(s) is performed before the test, comparison of residue and debris between inert [baseline test] and active items [test items];
g	Nature of any reactions by the Test Item;	Nature of any reactions by the Test Item;	Nature of any reactions by the Test Item;	Nature of any reactions by the Test Item;	Nature of any reactions by the Test Item as indicated by video and audio records;	Nature of any Test Item responses (donor and acceptor(s));
h	Imagery of the Test Item and the Test Setup before and after performing the test;	Imagery of the Test Item and the Test Setup before and after performing the test;	Imagery of the Test Item and the Test Setup before and after performing the test;	Imagery of the Test Item and the Test Setup before and after performing the test;	Photo Imagery of the Test Item and the Test Setup before and after performing the test;	Imagery of the Test Item and the Test Setup before and after performing the test;
i	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece; Number and depth of penetrations in fragment recovery panels (if used);	Nature and distribution of remains/residue and debris including: range, position, photographs, identification (as possible), and mass of each piece;
j	Meteorological data (wind speed, direction) during the test;	Meteorological data (wind speed, direction) during the test;	Meteorological data (wind speed, direction) during the test;	Meteorological data, in specific wind velocities and direction inside and outside the enclosure before the test, and any significant change in velocity/direction outside the enclosure (preferably well clear of the enclosure) during the test;	Meteorological data (wind speed, direction) during the test;	Meteorological data (wind speed, direction) during the test;
k	Indication of propulsion (video or other suitable means);	Indication of propulsion (video or other suitable means);	Indication of propulsion (video or other suitable means);	Indication of propulsion (video or other suitable means);	Indication of propulsion (video or other suitable means); Method of propulsivity measurement (e.g. mechanical devices, static/dynamic analysis, etc.) and technical justification supporting validity.	Indication of propulsivity (video or other suitable means);
l	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time.	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time.	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time.	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time. Perhaps something to filter out fuel burning sounds.	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time.	Audio and video records: A recording device shall be placed near the trial site to record all audio and enable correlation between visible events and indicated time. Means to differentiate these audible/visual events between the donor and acceptor.
m	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.	Suitable Blast or overpressure gauges should be positioned around the test item to record pressure-time history with a record of gauge location and height.
n	**Witness plates and screens** as a measure of projection severity (optional); Photographs of witness plates and screens (if used);	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).	Witness plates and screens (optional) as a measure of projection severity; Photographs of witness plates and screens (if used).
o	**A complete data record** shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.	A complete data record shall be compiled to include pressure, sound, imagery, fragmentation, debris and propulsion information.
p						

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OBSERVATIONS & RECORDS

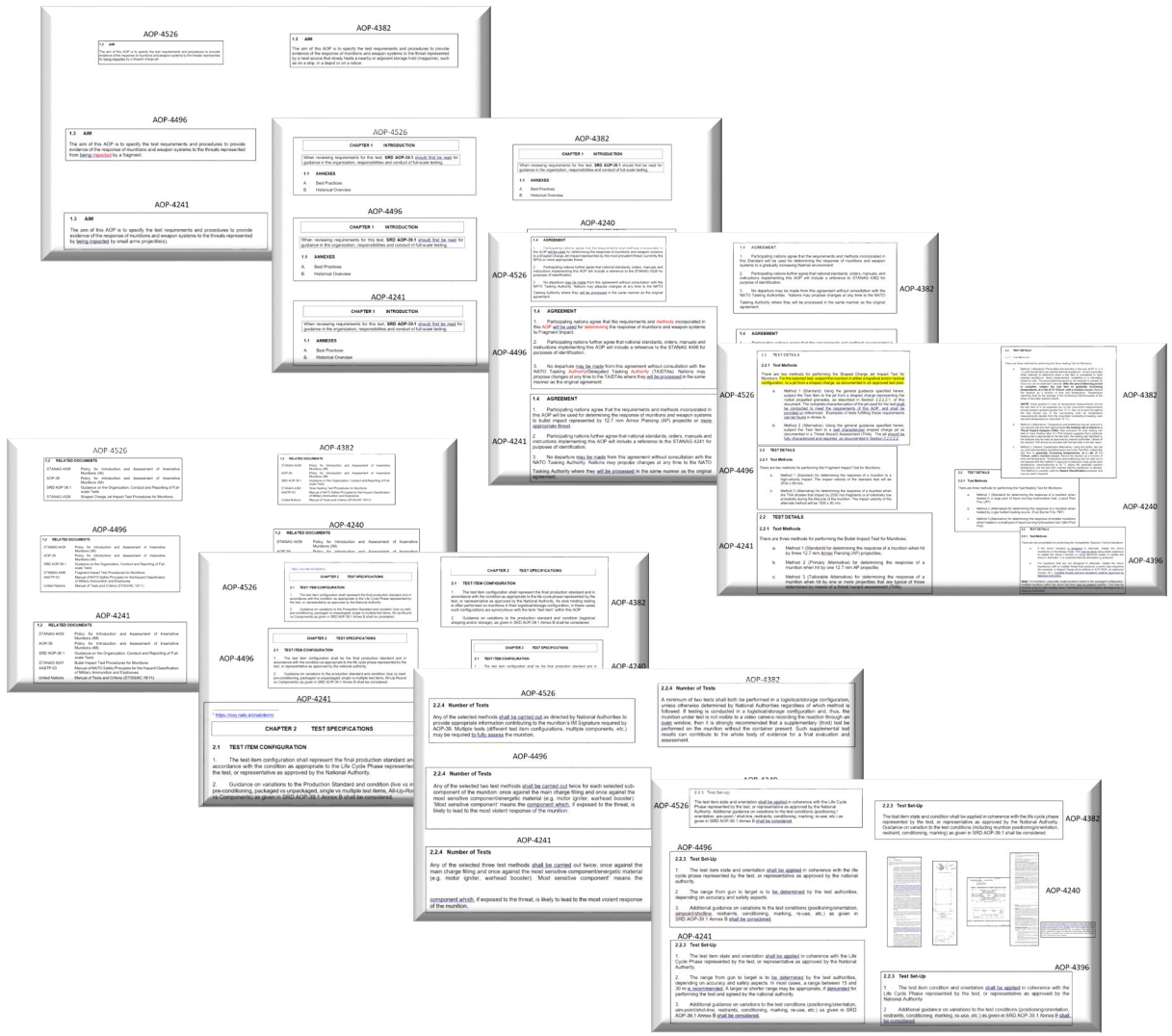
- ***Revised* Observations & Records Comparison Chart**
  - An organized matrix of (a-o) Ob's & Rec's
  - Only contains requirements found in AOPs
  - Consistent, Readable, Accurate, Clarified
  - Enabled efficient/effective comparison of section (i.e. 2.4).

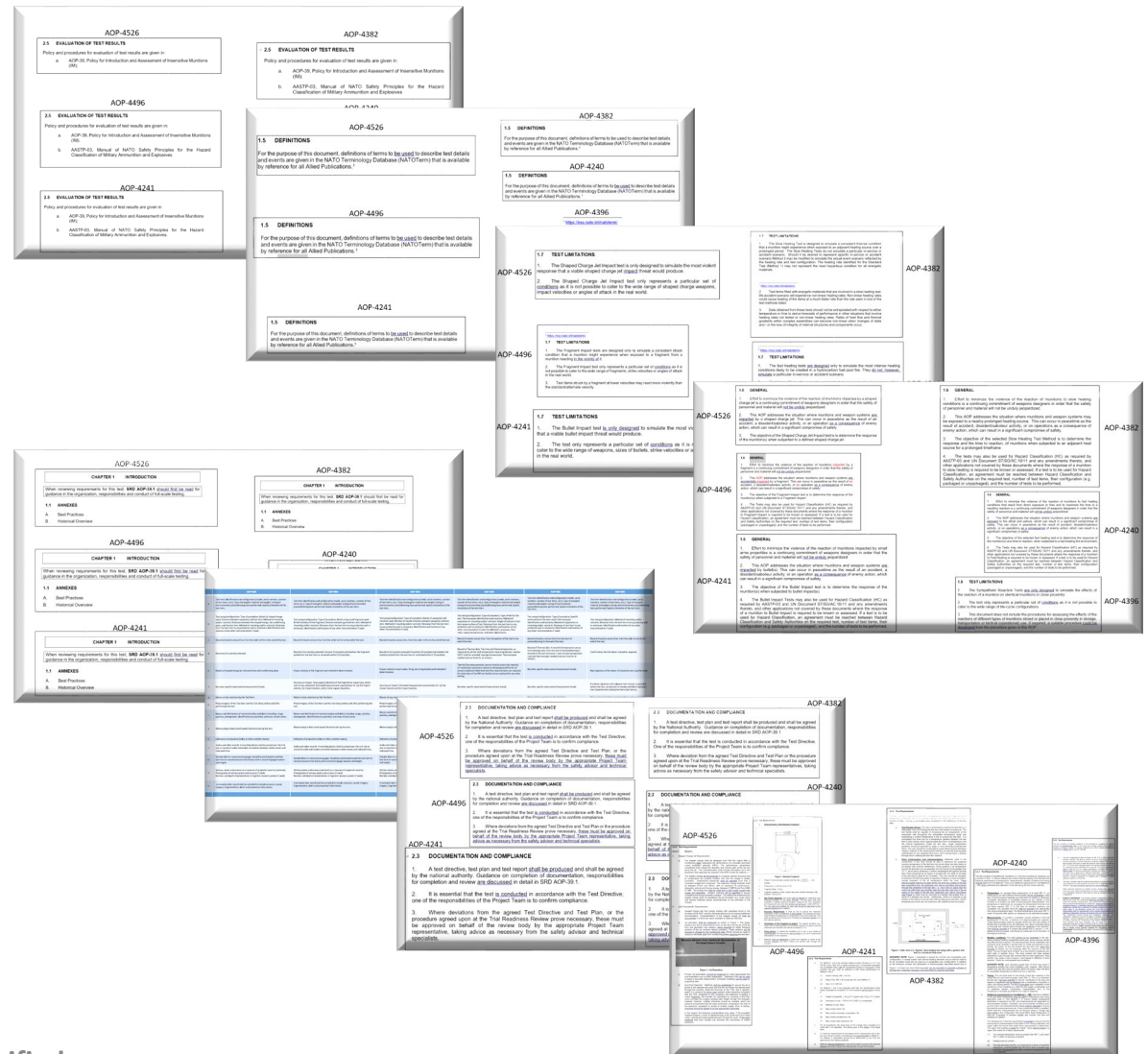
- **2.4 Observations & Records**
  - Custodians currently transferring content directly to respective AOPs in NATO format



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Next Meeting → Comparing Each Test AOP (Section-by-Section)





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Conclusions

NATO CNAD AC/326 CASG SG/B - IM Test AOP Standardization WG

- Updating NATO IM Test Doctrine Portfolio:
  - ✓ Ensuring *consistency* amongst the test documents
  - ✓ Common changes made to AOPs:
    - *Textual changes* for clarity of intent
    - *Outline structural revision* to improve:
      - Readability
      - Flow logic
      - Consistency
    - *Match the latest NATO AOP Format* (provided by MSIAC)
  - ✓ Unique changes made to AOPs:
    - To ensure technical metrics are accurately supported
  - ✓ Transferring some information from each AOP to SRD
    - Unnecessary, Additional , Guidance
  - ✓ Ultimately each of the 6 updated IM Test AOPs
    - Will undergo a *Version Update*
    - Will NOT undergo Ratification

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Conclusions

- Synchronizing changes made to AOPs with other related / supporting documents
  - AOP-39, AOP-39.1 (SRD), AOP-IM/HC, etc.
    - AOP-39 & SRD will also undergo Version Change only
- Captured and addressed necessary justifications for changes made, by deliberating over:
  - Terms
  - Definitions
  - Intentions
- Created a Record of Memorandum to record these changes / justifications for future portfolio revisions
- Utilized Comparison Chart for ease of evaluating, comparing, and cross-analyzing topics amongst the documents
  - We will retain / maintain these comparison charts in our records to make it easier for us during our next revision of the AOPs in the future.
    - Improve our revision process
    - Helped retain old information and reasons for changes
    - Perhaps even users may find these useful when running suite of IM tests.
      - Can use as ‘Reference Table’

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References

1. K. Tomasello, C. Jacq, E. Baker, M. Sharp, 'NATO AC326 SG/B: Ammunition Systems Design and Assessment – IM Test STANAG updates', IMEMTS, Silken Al-Andalus Palace Hotel, Seville, Spain, Oct 21-24, 2019.
2. D. Pudlak, B. Fuchs, 'Guidance on Test Item Configurations for Sympathetic Reaction Testing in Accordance with AOP-4396', IMEMTS, Silken Al-Andalus Palace Hotel, Seville, Spain, Oct 21-24, 2019.
3. AOP-39, Policy for Introduction and Assessment of Insensitive Munitions (IM), 2019.
4. AOP-39.1, STANDARDS RELATED DOCUMENT, GUIDANCE ON THE ORGANISATION, CONDUCT AND REPORTING OF FULL SCALE TESTS, EDITION A VERSION 1, MAY 2018.
5. North Atlantic Council, STANAG 4240 – Fast Heating, Munitions Test Procedures, Nov 2018.
6. North Atlantic Council, STANAG 4241 – Bullet Impact, Munitions Test Procedures, Nov 2018.
7. North Atlantic Council, STANAG 4382 – Slow Heating, Munitions Test Procedures, Mar 2020.
8. North Atlantic Council, STANAG 4396 – Sympathetic Reaction, Munitions Test Procedures, Dec 2020.
9. North Atlantic Council, STANAG 4496 – Fragment Impact, Munitions Test Procedures, March 2019.
10. North Atlantic Council, STANAG 4526 – Shaped Charge Jet Impact, Munitions Test Procedures, Nov 2018.

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Thank You

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Improved Particle Size Reduction & Evaluation Technology for Energetics at HSAAP

IMEMTS 2021

Dr. Jacob Morris*, Dr. Neil Tucker, Matt Hathaway, Mike Ervin
BAE Systems, Holston Army Ammunition Plant, Kingsport, Tennessee, United States
7 April 2021



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BAE SYSTEMS

Overview

- The final particle size of traditional crystalline explosives (RDX/HMX) at Holston is controlled through a number of technologies:
  - Solvent Recrystallization
  - Wet-grinding
  - Fluid Energy Milling

- Evaluation of newer technologies could lead to reduced processing time and better control of process

- Technology requirements:
  - Ability to safely process explosives
  - Improvement in processing time/control vs. legacy technology
  - Ability to meet particle size/morphology requirements to serve as drop-in replacement
  - Cost to develop / implement technology

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RDX/HMX Particle Size Technology

RDX/HMX Particle Size

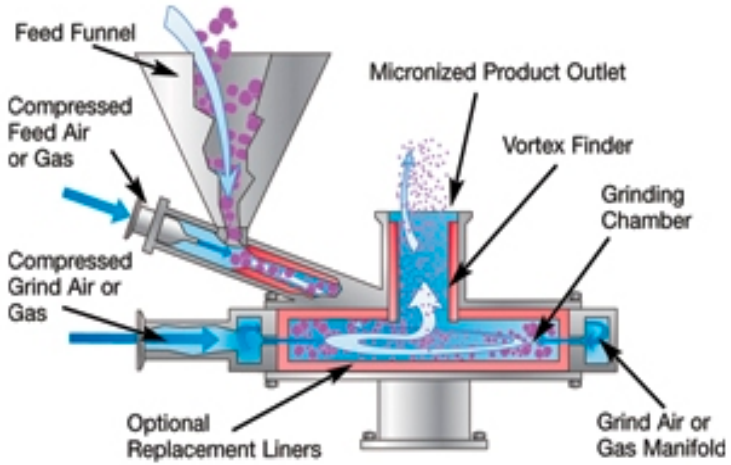
Sub-micron (New class)	2-8 µm (FEM)	10-50 µm (Cl 2 & 5)	150-300 µm (Cl 1, 6, 7)	300-600 µm (Class 3)	>600 µm (Class 4)

Solvent based Recrystallization

Traditional:

Wet Grinding

Fluid Energy Milling



Fluid Energy Mill



Cowles Grinder



Recrystallization Vessel

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RDX/HMX Particle Size Technology

RDX/HMX Particle Size

Sub-micron (New class)	2-8 µ (FEM)	10-50 µ (Cl 2 & 5)	150-300 (Cl 1, 6, 7)	300-600 µ (Class 3)	>600 µ (Class 4)

Traditional:

- Solvent based Recrystallization
- Wet Grinding
- Fluid Energy Milling

Potential:

- Non-traditional Solvent based Processing
- IKA Grinding
- Novel Milling Technology

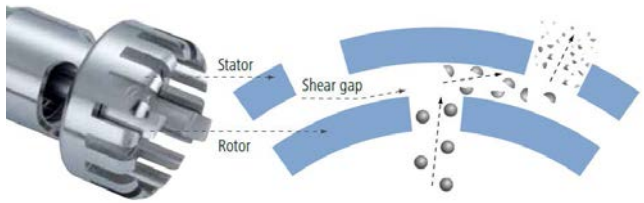


IKA Grinder

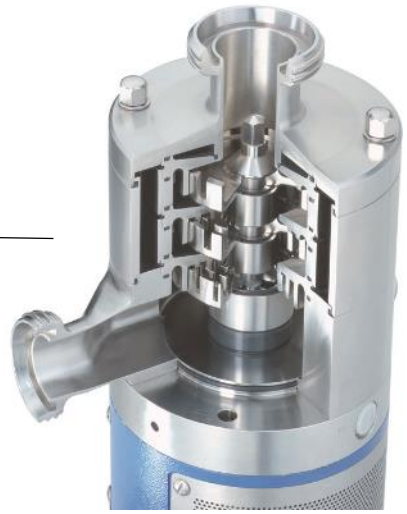
IKA Grinder: Evaluation for Replacement of Traditional Wet-Grinding Technology

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RDX/HMX IKA Particle Size Reduction Technology



High-Shear Dispersion



3-Stage Rotor-Stator Head

- Slurry-based particle size reduction technology
- Three–stage rotor-stator heads in series
  - Multiple size heads allow targeted PSD reduction
- Ability to vary power setting, recirculate slurry, and multiple units in series allows for further targeted PSD reduction
- Flexible low-cost technology
- Throughput: 500 L/hr (pilot unit)

IKA Grinder: High shear wet-grinding technique for safe particle size reduction of energetic crystals

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Case Study: HMX Class 5 (Pump Ground)

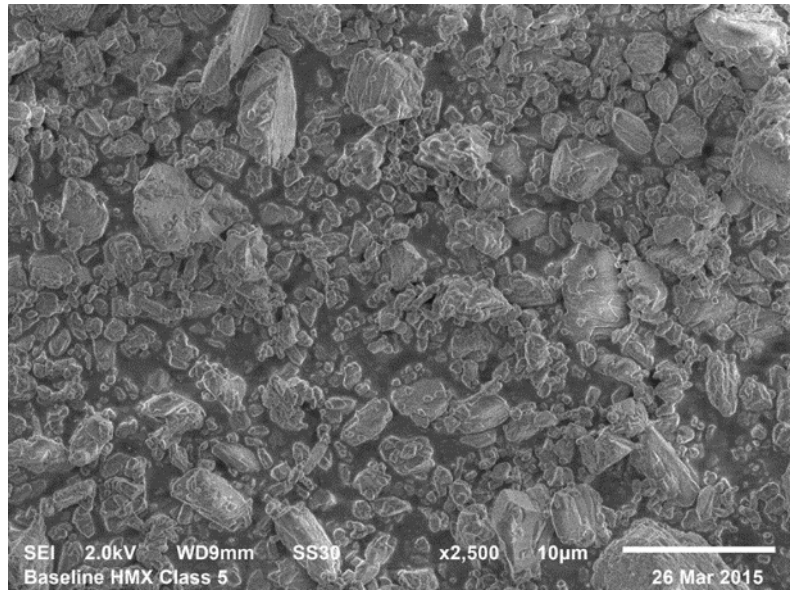


Pump Grinder

- HMX Class 5 (Pump Ground) used in number of legacy HSAAP Products
  - Time / Energy inefficient process
  - Difficulty in reliably meeting targeted particle size

Particle Size Attributes:

Analytical Test	Value
Malvern, 10th Percentile	1.57 µm
Malvern, 50th Percentile	4.11 µm
Malvern, 90th Percentile	16.64 µm
BET Surface Area	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL



HMX Class 5

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Case Study: HMX Class 5 (Pump Ground)

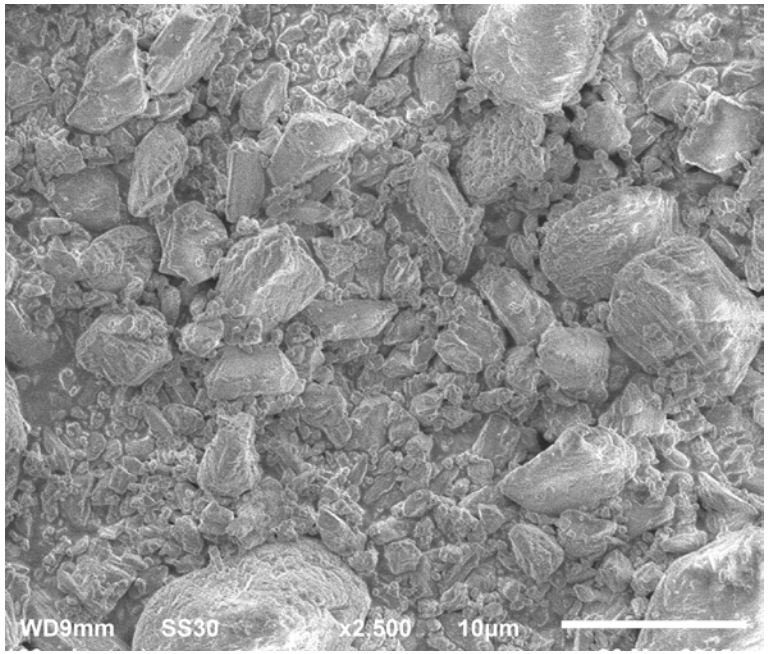


IKA Grinder

- Evaluation of **IKA Grinder** as replacement technology for pump grinder:
  - Reduction in processing time by >90%
  - Particle size/shape similar to pump ground material
  - Process to be fine-tuned in terms of concentration, power settings, recirculation time

Particle Size Attributes:

Analytical Test	IKA Ground	Pump Ground
Malvern, 10th Percentile	3.60 µm	1.57 µm
Malvern, 50th Percentile	7.10 µm	4.11 µm
Malvern, 90th Percentile	22.60 µm	16.64 µm
BET Surface Area	1.30 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



HMX Class 5 with IKA Grinder

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Case Study: HMX Class 5 (Pump Ground)

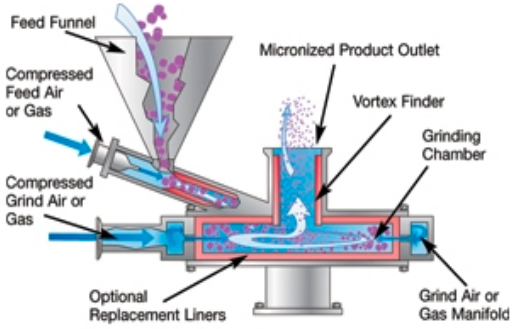
Evaluation of alternate particle size reduction technology to replace pump grinder:

- Fluid energy mill
- Crash precipitation from solvent
- Spray atomization
- Lab RAM
- Ultrasonic Treatment
- Roll Mill
- Cowles Grinder
- Fluidized bed mill
- Planetary ball mill
- Super critical CO_2

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Case Study: HMX Class 5 (Pump Ground)

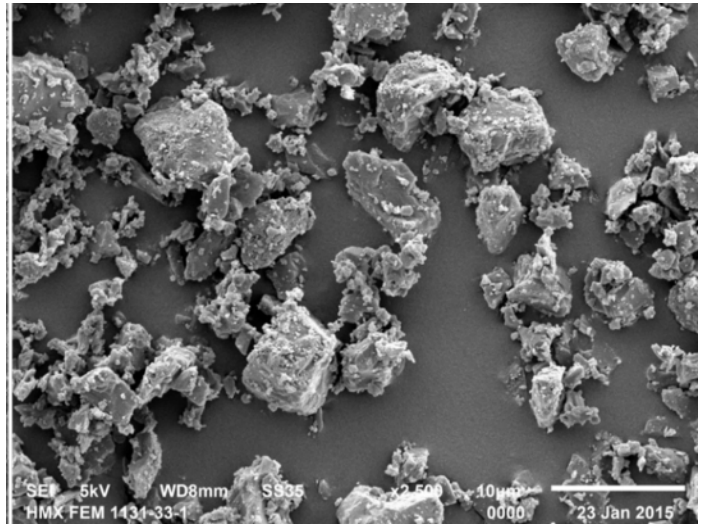
- Evaluation of FEM as replacement technology for pump grinder:
  - Mature particle size reduction technology at HSAAP
  - Particle size/shape similar to pump ground material with material slightly finer
  - Viable processing alternative with some downsides:
    - Dry milling vs. wet milling
    - No improvement in processing time



Fluid Energy Mill

Particle Size Attributes:

Analytical Test	FEM	Pump Ground
Malvern, 10th Percentile	1.64 µm	1.57 µm
Malvern, 50th Percentile	3.49 µm	4.11 µm
Malvern, 90th Percentile	7.35 µm	16.64 µm
BET Surface Area	3.24 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



HMX Class 5 with FEM

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Case Study: HMX Class 5 (Pump Ground)

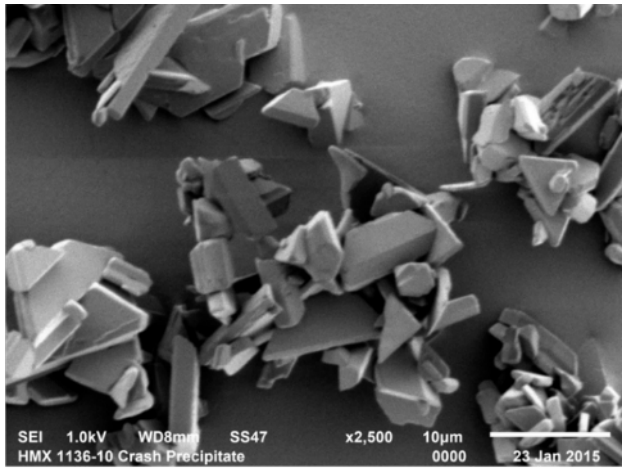
- Evaluation of Solvent Crash Precipitation as replacement technology for pump grinder:
  - Evaluation of batch/continuous processing
  - Particle size slightly larger than pump ground material w/ different morphology
  - Viable processing alternative with potential downsides:
    - Potential presence of impact-sensitive HMX polymorphs
    - Ability to control particle size at production scale



Recrystallization Vessel

Particle Size Attributes:

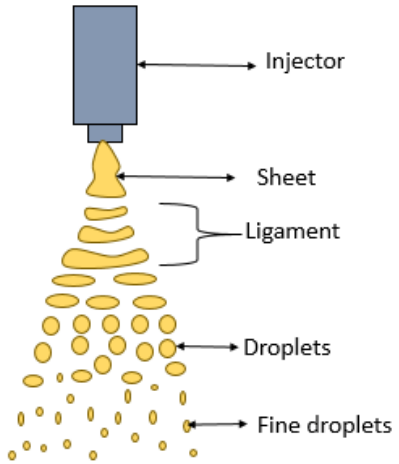
Analytical Test	FEM	Pump Ground
Malvern, 10th Percentile	3.47 µm	1.57 µm
Malvern, 50th Percentile	7.08 µm	4.11 µm
Malvern, 90th Percentile	16.02 µm	16.64 µm
BET Surface Area	1.40 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.82 g/mL	1.91 g/mL



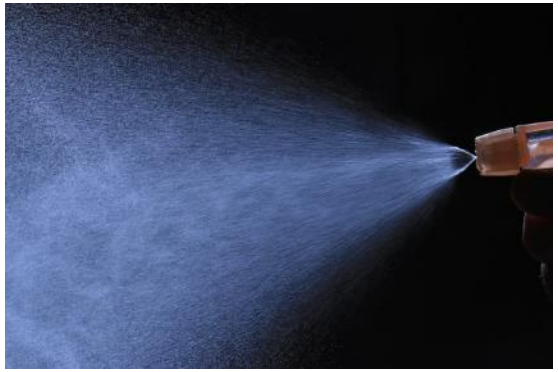
HMX Class 5 through Crash Precipitation

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Case Study: HMX Class 5 (Pump Ground)



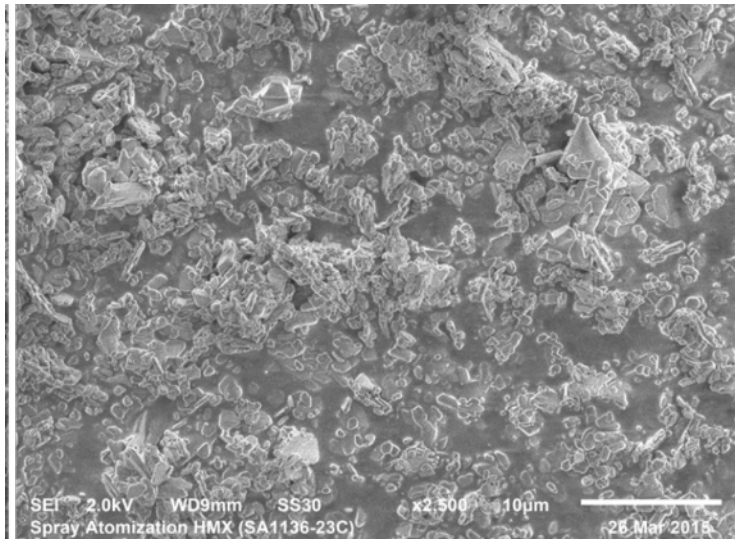
- Evaluation of Spray Atomization as replacement technology for pump grinder:
  - Use of solvent/anti-solvent spray atomization process
  - Particle size/shape similar to pump ground material
  - Viable processing alternative with potential downsides:
    - Unreliable control on particle size at laboratory scale
    - Large amount of solvent recovery required at production scale



Spray Atomization

Particle Size Attributes:

Analytical Test	FEM	Pump Ground
Malvern, 10th Percentile	1.80 µm	1.57 µm
Malvern, 50th Percentile	4.18 µm	4.11 µm
Malvern, 90th Percentile	11.91 µm	16.64 µm
BET Surface Area	3.82 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



HMX Class 5 through Spray Atomization

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Case Study: HMX Class 5 (Pump Ground)

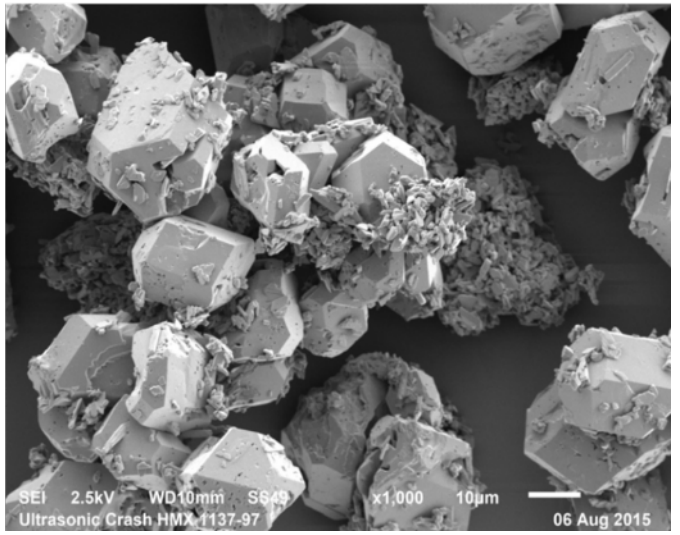
- Evaluation of Ultrasonic Probe as replacement technology for pump grinder:
  - Use of ultrasonic probe in combination with solvent/anti-solvent process
  - Particle size/shape larger than pump ground material
  - Significant engineering required to overcome processing hurdles:
    - Significant particle size growth if material filtered slowly
    - Fine-tuning of processing parameters and probe required



Ultrasonic Probe

Particle Size Attributes:

Analytical Test	FEM	Pump Ground
Malvern, 10th Percentile	9.59 µm	1.57 µm
Malvern, 50th Percentile	21.26 µm	4.11 µm
Malvern, 90th Percentile	38.05 µm	16.64 µm
BET Surface Area	0.52 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.88 g/mL	1.91 g/mL



HMX Class 5 through Ultrasonic Probe

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Case Study: HMX Class 5 (Pump Ground)

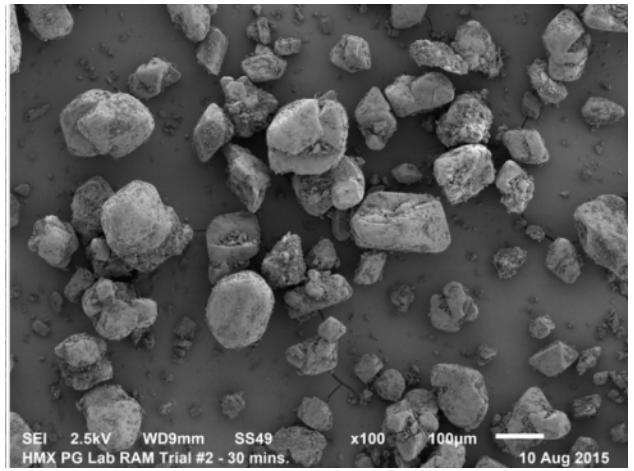
- Evaluation of LabRAM as replacement technology for pump grinder:
  - Use of resonant acoustic energy to cause particle size attrition
  - Not a viable option for Pump grinder replacement
    - Further evaluation of LabRAM with solvent/antisolvent system a possibility



LabRAM

Particle Size Attributes:

Analytical Test	FEM	Pump Ground
Malvern, 10th Percentile	5.67 µm	1.57 µm
Malvern, 50th Percentile	67.32 µm	4.11 µm
Malvern, 90th Percentile	199.01 µm	16.64 µm



HMX Class 5 through LabRAM

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Case Study: LLM-105

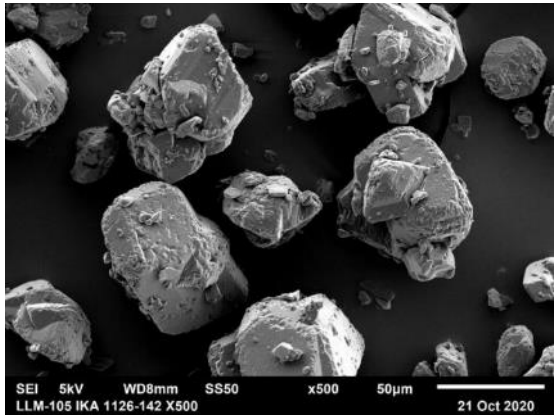
- Goal: Reduction in LLM-105 particle size through wet-milling technology
  - Cowles Grinder
  - IKA Grinder
- Targeting d_{50} of ~60 microns
- While both technologies successful, IKA grinder was >90% faster to meet particle size target



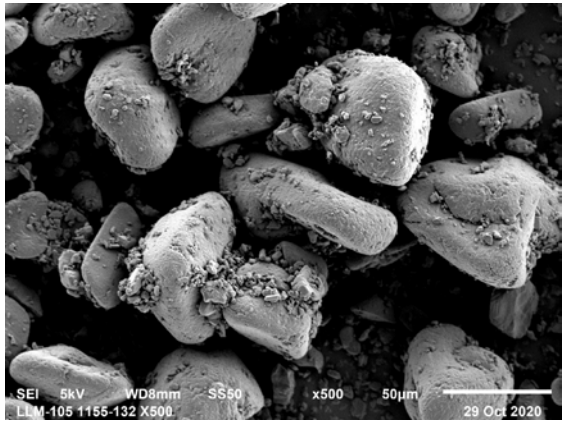
Cowles Grinder

Particle Size Attributes:

Analytical Test	IKA Grinder	Cowles Grinder
Malvern, 10th Percentile	29.01 µm	7.62 µm
Malvern, 50th Percentile	57.16 µm	48.01 µm
Malvern, 90th Percentile	98.74 µm	92.01 µm



LLM-105 (IKA)



LLM-105 (Cowles)

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Summary and Path Forward

- Use alternate technologies to replace HMX Class 5 (Pump Ground)
  - IKA Grinder shows good promise as newer wet-milling technology
  - FEM HMX also shows promise and has production scale maturity

- Path Forward (IKA Grinder)
  - Further evaluation of alternate materials / particle size classes
  - Evaluation of IKA-ground material is various formulations
  - Further development of technology from pilot to production scale

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Acknowledgements

- BAE Systems OSI – Holston Army Ammunition Plant
  - Mr. Brian Alexander
  - Ms. Kelly Smith
  - Ms. Alice Meadows
  - Mr. Joseph Renfro
  - Mr. Gary Sizemore
  - Mr. Todd Dye
  - Mr. Tracy Kelly

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Thank You



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Influence of Ageing on an IM Signature

Anti-Aircraft Missile Propulsion Unit

Quentin WEISSE

DGA Missiles Testing
BP 80070
33166 Saint-Médard-en-Jalles Cedex, France

@ : quentin.weisse@intradef.gouv.fr

Hervé Benard

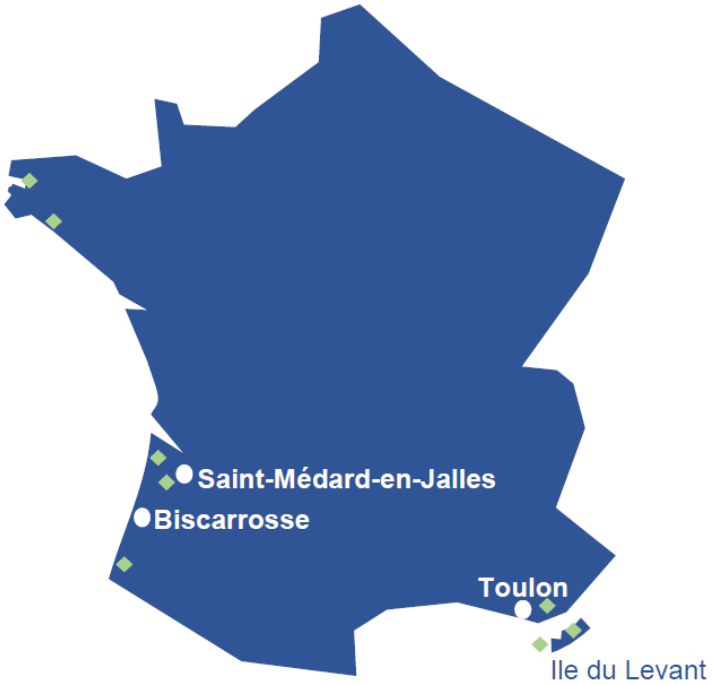
Organization for Joint Armament Co-operation (OCCAR)
106 Av. Marx Dormoy
92120 Montrouge

@ : herve.benard@occar.int

DGA
DIRECTION GÉNÉRALE
DE L'ARMEMENT

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DGA Missiles Testing



50 YEARS OF EXPERIENCE AND EXPERTISE

3 main sites

- Gironde
- Landes

test and measurement ship « Le Monge »

- Méditerranée

950 staff DGA Missiles testing



Key figures (/year)

- **120 M€** Sales
- **80 M€** Invests
- **~400** Missiles and weapon systems tested
- **~250** Targets used
- **~100** Ground tests

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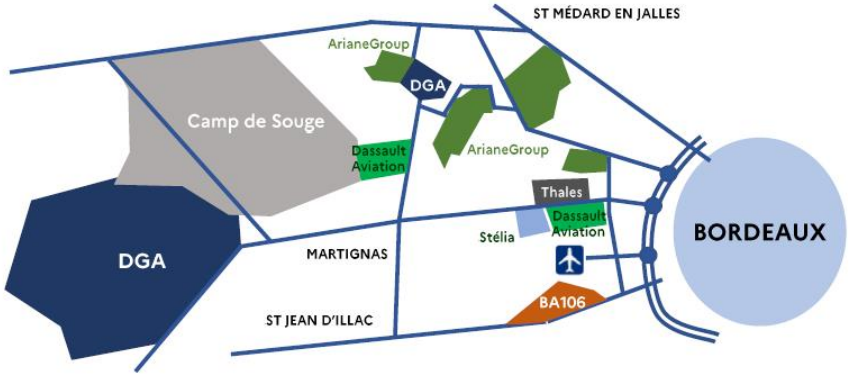


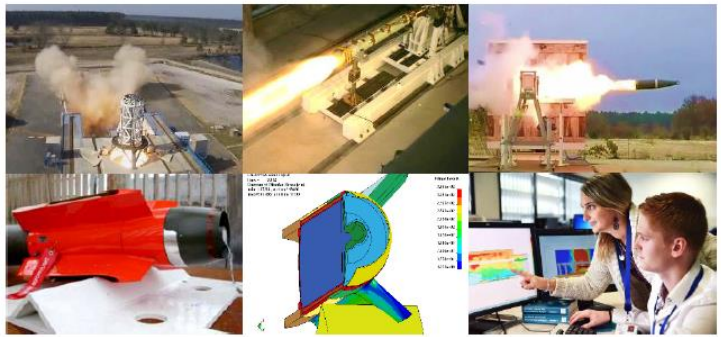
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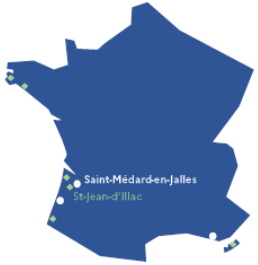
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GIRONDE SITE



2 locations
St Médard-en-Jalles & St Jean d'Illac

10 Test Benches

- In **different configurations**
- **Safety** test areas
- A unique **altitude flight** assessment facility in Europe

ACTIVITIES

TACTICAL PROPULSION
Ageing programs
Upstream studies and technologies
Amont and technologies

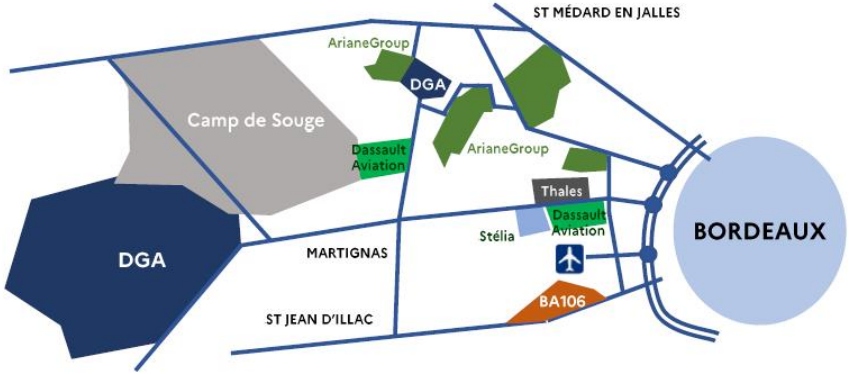
EXPERTISE
Assistance for project management
Support for testing
Referent experts

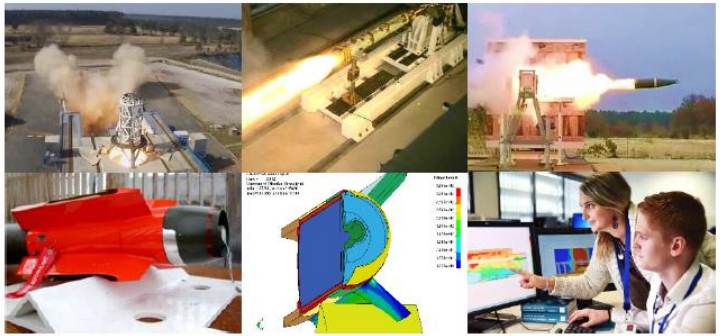
HEAVY PROPULSION
MSBS (M45 ; M51.x)
Spatial (ARIANE, VEGA, ARTA,...)

VULNERABILITY/SECURITY
Essential step before operational use by the forces
Unique French test center labeled NATO

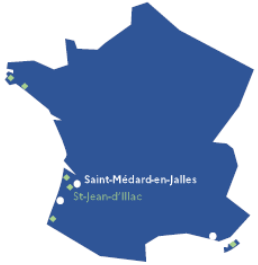
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Munitions Performances and Ageing Assessment

DGA MT Scope:

- Prime contractor for entire or partial ageing programmes (definition, thermal cycle, test, analysis, etc)
- Assessment of the service life duration of pyrotechnical devices
- Expertise and support for Armament Programs



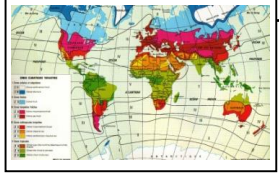
Tested Items:

- Solid Rocket Motors
- Warheads
- Pyrotechnical devices (SAU, batteries, etc)
- Complete missiles

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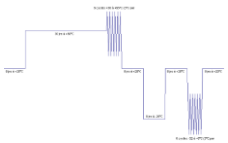
Reference Documents
STANAG 4370, AETCP-230, …

Threat Health Analysis



Ageing Program Definition

Thermal Cycles



Mechanical Environment Test



Ageing Program Realization

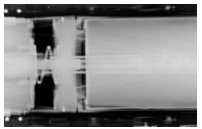
Natural or Accelerated Ageing



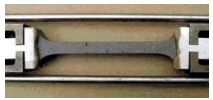
Non-Destructive Inspection

(X-ray, Fluoroscopy, Endoscopy, …)





Dissection



Evaluation of the Aged Specimens

Bench Firing (-50 à +80°C)





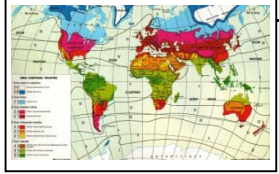
IM Tests



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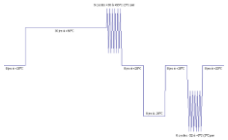
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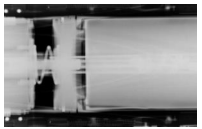
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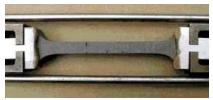


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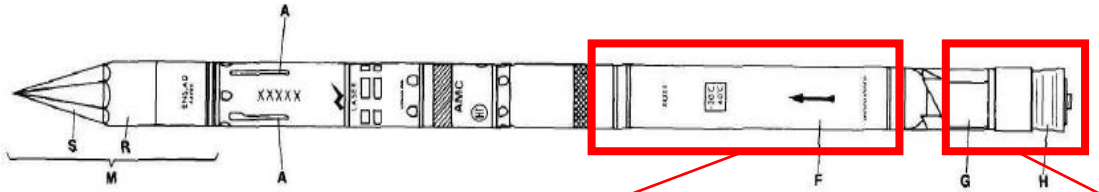
IM Tests

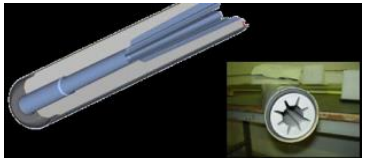


⇒ **First Results on a Anti-Aircraft Missile Propulsion Unit…**

Presentation of the Munition

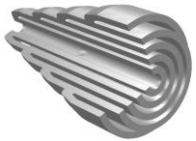
Anti-Aircraft Missile Propulsion Unit





Rocket Motor

- Butalane 68/20 Propellant
- Composite structure (kevlar/epoxy) with humidity barrier



Launch Motor

- Solventless Propellant (EDBP)
- Printed block enclosed in a metallic structure

⇒ **Different types of ageing phenomenology**

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Main Ageing Phenomenologies
Launch Motor

Solventless Propellant (EDBP)

- Thermolysis
  - ⇒ **Decomposition of Nitrate Esters**
  - ⇒ **Consumption of stabilizer**
- Migration of Plasticizer
  - ⇒ **Weakened material**
- Migration of Nitroglycerin
  - ⇒ **Exudation**
  - ⇒ **Waste of potential**



Consequences

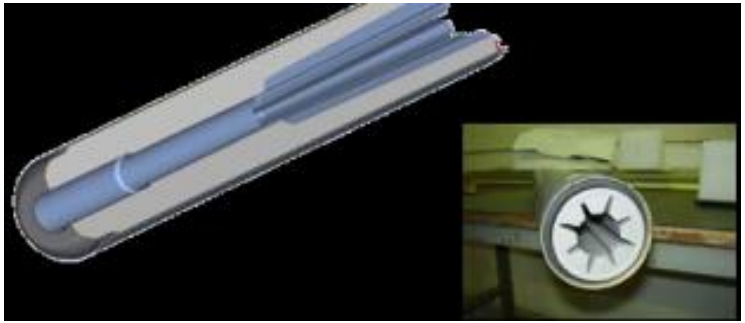
- Impact on the Mechanical Behavior and Safety
- Potential loss in ballistic performances

Main Ageing Phenomenologies

Rocket Motor

Composite Propellant (Butalane with PBHT binder)

- Thermo-oxidation
  - ⇒ **Binder Hardening**
  - ⇒ **Weakened material**
  - ⇒ **Higher cracking risk**
- Hydrolysis
  - ⇒ **Propellant Softening**



Consequences

- Impact on the mechanical behavior
- Safety risk in case of cracking or loss of mechanical property
- If softening, small impact on ballistic performances

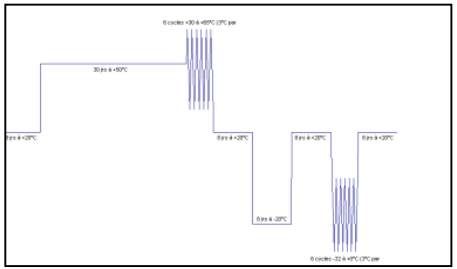
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Accelerated Ageing Program



Objectives:

- Predict the lifespan of amunition regarding its operational life
- Control of the operational safety and performances



Principles:

- Replicate the life of the munition
- Definition of climatic and mechanical cycles

Application to the Anti-Aircraft Missile Propulsion Unit:

- Accelerated Ageing of 23 or 24 years
- Representative ageing for the Ejector more important than for the Rocket Motor

γ_{10} **EDBP propellant** > γ_{10} **butalanes**

Control After Ageing

Non-Destructive Controls:

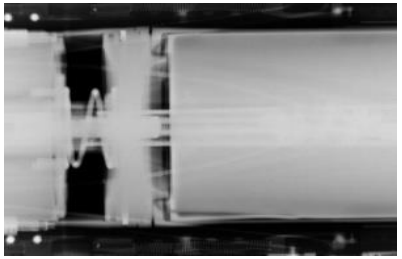
- Integrity of the specimen before firing
- State of the filling contour

Dissection:

- Material evolution
- Reliability of the architecture

Bench Firing:

- Validation of the ballistic performances
- Control of the security of firing and the aptitude to complete mission







And for IM tests ?

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NATO References and Reaction Types

Policy for Introduction and Assessment of Insensitive Munitions (IM)	**STANAG 4439 Ed. 4** AOP-39 Ed. D V. 1
Fast Heating (FH)	**STANAG 4240 Ed. 3** AOP-4240 Ed. A V. 1
Slow Heating (SH)	**STANAG 4382 Ed. 3** AOP-4382 Ed. A V. 1
Bullet Impact (BI)	**STANAG 4241 Ed. 3** AOP-4241 Ed. A V. 1
Fragment Impact (FI)	**STANAG 4496 Ed. 2** AOP-4496 Ed. A V. 1
Shaped Charge Jet Impact (SCJI)	**STANAG 4526 Ed. 3** AOP-4526 Ed. A V. 1
Sympathetic Reaction (SR)	**STANAG 4396 Ed. 3** AOP-4396 Ed. A V. 1
Safety Drop	**STANAG 4375 Ed. 3**



Type I – Detonation



Type II – Partial Detonation



Type III – Explosion



Type IV – Deflagration



Type V – Burn



Type VI – No Reaction

Initial IM Signature

Factory Fresh

	Ejector	Rocket Motor
Fast Heating	V	V
Slow Heating	III	(I/II)
Bullet Impact	III	IV
Fragment Impact	(I/II)	(I/II)
Shaped Charge Jet Impact	(I/II)	(I/II)
Sympathetic Reaction	(I/II)	(I/II)

(X): No test performed (type I/II expected)

Drop Test	NR	NR

Initial IM Signature

Factory Fresh

	Ejector	Rocket Motor
Fast Heating	V	V
Slow Heating	III	(I/II)
Bullet Impact	III	IV
Fragment Impact	(I/II)	(I/II)
Shaped Charge Jet Impact	(I/II)	(I/II)
Sympathetic Reaction	(I/II)	(I/II)

(X): No test performed (type I/II expected)

Drop Test	NR	NR

⇒ **Great Interest to observe the evolution of FH, SH, BI and DT results with Ageing…**

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Influence of Ageing on the IM Signature

Fast Heating on the complete propulsion unit

Factory Fresh	After Ageing
Complete propulsion system in its tactical configuration Combustion of the propulsion unit after 6 min 35 s Short distance projections No propulsion effect measured ⇒ **Type V**	**Natural Ageing > 20 years** Similar results compared with the Factory Fresh item ⇒ **Type V**



No influence on the FH Test Results for the Ejector and the Rocket Motor

Influence of Ageing on the IM Signature

Slow Heating on the Ejector

Factory Fresh	After Ageing
SH Test done on the entire Propulsion Unit Reaction of the Ejector led to the projection of the Rocket Motor far from the Hoven Extensive fracture on the case of the Ejector Low Pressure wave throughout the test area Outside combustion ⇒ **Type III**	**Natural Ageing > 15 years** SH Test carried out on the Ejector with an inert Rocket Motor Similar results compared with the Factory Fresh item ⇒ **Type III**



No influence on the SH Test Results for the Ejector

Influence of Ageing on the IM Signature

Slow Heating on the Rocket Motor

Factory Fresh	After Ageing
SH Test done on the entire Propulsion Unit in its tactical configuration Reaction of the Ejector led to an interuption of the heating cycle No reaction observed for the Rocket Motor **=> Type I/II expected**	**Natural Ageing > 15 years** SH Test carried out on the Rocket Motor with an inert Ejector in its tactical configuration Violent Reaction of the Rocket Motor Important fracture of the case Shock wave with an important magnitude (partial detonation) ⇒ **Type II**



Consistent with the expected reaction for the Rocket Motor

No improvment of the reaction with Ageing

Influence of Ageing on the IM Signature

Bullet Impact on the Ejector

Factory Fresh	After Ageing
Complete propulsion system in its tactical configuration Impact Velocity: 847 m/s Extensive fracture on the case of the Ejector Low Pressure wave throughout the test area Outside combustion from a part of the propellant Undamaged rocket motor ⇒ **Type III**	**Equivalent Age > 20 years** Complete propulsion system in its tactical configuration Impact Velocity: 844 m/s Similar results compared with the Factory Fresh item ⇒ **Type III**



No influence on the BI Test Results for the ejector:

- Valid only for direct shock ignition phenomenology

Different conclusion in case of lodged fragment?

- To explore in the future

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Influence of Ageing on the IM Signature

Bullet Impact on the Rocket Motor

Factory Fresh	After Ageing
Complete propulsion system in tactical configuration Impact Velocity: 851 m/s Separation of the case in 2 large pieces Low Pressure wave throughout the test area Long distance projection of the Ejector No measurement of propulsion ⇒ **Type IV**	**Equivalent Age > 20 years** Complete propulsion system in tactical configuration Impact Velocity: 840 m/s Similar results compared with the Factory Fresh item Phenomenology of propulsion measured ⇒ **Type IVp**



No influence on the BI Test Results for the Rocket Motor:

- Valid only for direct shock ignition phenomenology

Different conclusion in case of lodged fragment?

- To explore in the future

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Influence of Ageing on the Drop Test Results

Complete Missile

Factory Fresh	After Ageing
Complete missile in logistical container ⇒ **No Reaction** Complete missile in Tactical Tube (worst configuration) ⇒ **No Reaction**	**Equivalent Age > 20 years** Complete propulsion unit with inert warhead Damages similar to the Factory Fresh test ⇒ **No Reaction**



No influence on the Drop Test Results for the Rocket Motor and the Ejector

Conclusion

First Results about the influence of Ageing on an IM Signature

- Old program with out-of-date IM objectives
- Entire IM Spectrum not covered

Good results on FH, SH and BI Tests for Composite and Double-Base Propellant

- BI Results valid only for direct shock ignition phenomenology

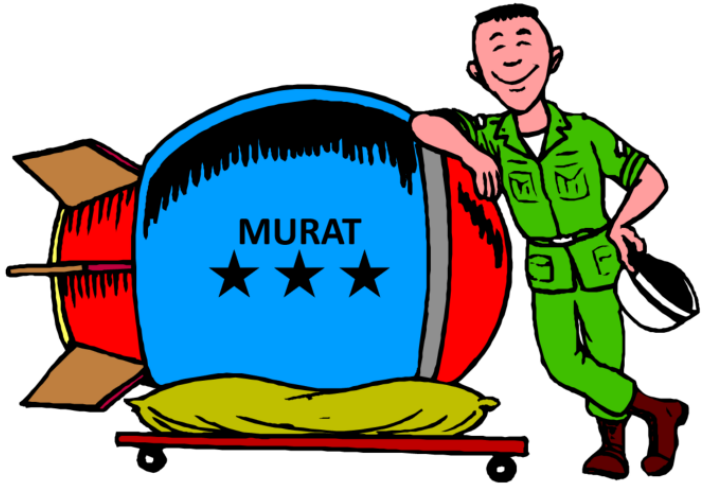
Perspective to do a complete analysis on a more recent program

- Specimen collection at the end of its operational life

MINISTÈRE
DES ARMÉES

Liberté
Égalité
Fraternité

Questions ?



DGA
DIRECTION GÉNÉRALE
DE L'ARMEMENT

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U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Continuous Reactors for Energetic Materials Synthesis

Joseph Rheinhardt, PhD

Research Scientist III

CS Squared, LLC

Approved for Public Release; Distribution is unlimited.

07 April 2021

AGENDA

- **Answering the "When" of Flow**
  - Advantages of Flow Chemistry: The Three C's
    - **C**hemistry
    - **C**ontrol
    - **C**ost
  - Screening Reactions for Continuous
    - Calabrese and Pissavini Screening Framework
    - Roberge Reaction Classification Scheme
    - Reaction/Reactor Selection Matrix

- **Answering the "What" of Flow**
  - Transformers and Generators
  - Survey of Commercial Off-The-Shelf Apparatus
    - Continuous Reactors
    - Continuous Crystallizers and Filters
    - Ancillary Equipment
      - Spectroscopic Tools
      - Pumps
      - Separators

- **Summary and Additional Resources**

Answering the “When” of Flow

ADVANTAGES OF FLOW: THE THREE C's

- **Chemistry**
  - Safety: Smaller scale; No headspace; No accumulation of reactive or toxic intermediates
  - Expansion of the Reaction Space: "Forbidden" reactions are feasible in flow; Reactions in Novel Process Windows (high temperature, high pressure, increased concentration/solvent-free, explosive or thermal runaway regimes)

- **Control**
  - Seamless Scale Up: Scale up (numbering up) is faster and more reliable
  - Versatility and Flexibility: Customizable and adjustable equipment; Ease of switching products; Flexibility to turn throughput up or down
  - Increased space–time yield (increased throughput in smaller footprint)
  - Steady-state, continuous process feedback
  - Quality by Design (QbD)/Quality by Control (QbC)

- **Cost**
  - Less energy, water, and raw material costs (solvents, cleaning fluid)
  - Lower labor cost
  - Less capital depreciation
  - Increased product yield and quality

Jensen, K. *Microreactors in Discovery and Development*, *PoaC Symposium, **2013***.
Calabrese, G. S.; Pissavini, S. *AIChE Journal,* **2011**, *57(4)*, 828-834.

SCREENING REACTIONS FOR CONTINUOUS

Pressures exceed reactor capability? Temperatures exceed reactor capability? Chemistry not compatible with reactor? Equilibrium reactions?	Likely no benefit and/or not possible	Increasing applicability
Solid precipitates? Very slow kinetics? Solid reactants or catalysts? Gaseous reactants? Homogeneous catalysts?	Possible benefits, but with technical challenges	
Gas evolution? Reaction benefits from pressure > 120 Pa Unstable intermediates? Fast kinetics? Highly toxic reactants or byproducts? Reactions requires or benefits from T < -10 °C? Rapid mixing required? Highly exothermic? Over-reaction possible? Requires precise stoichiometric control?	Likely to benefit	

Calabrese, G. S.; Pissavini, S. *AIChE Journal,* **2011**, *57(4)*, 828-834.

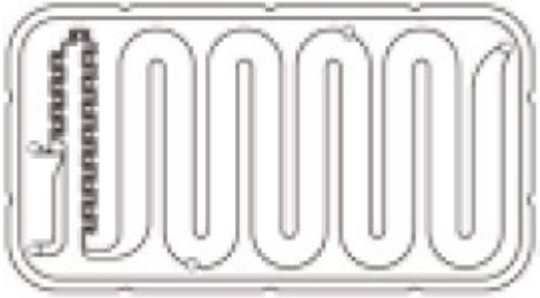
ROBERGE REACTION CLASSIFICATION SCHEME

- **Type A reactions**
  - Very fast, typically < 1 s
  - Controlled by diffusion and mixing
  - Improved mixing/heat exchange may increase yield
- **Type B reactions**
  - Rapid reactions, typically 10 s to 30 min
  - Kinetically controlled – rate may be accelerated by increased temperature, pressure, or concentration
- **Type C reactions**
  - Slow reactions, > 30 min to hours
  - Involve potential hazards such as autocatalysis or thermal accumulation
  - Kinetics make reactions suitable for batch, but continuous offers improved safety or product quality
- **Type D reactions**
  - Reactions that are not A, B, or C
  - Should be intensified to at least Type C to be done in flow

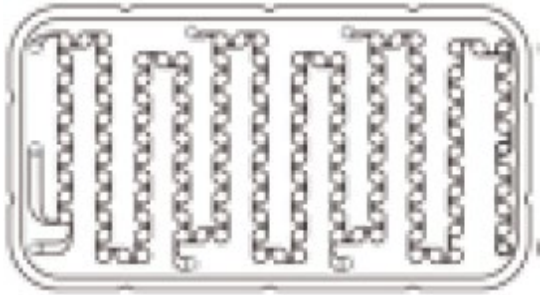
Plouffe, P.; Macchi, A .; Roberge, D. M. *Org. Proc. Res. Dev.,* **2014**, *18*, 1286-1294.

REACTION/REACTOR MATRIX

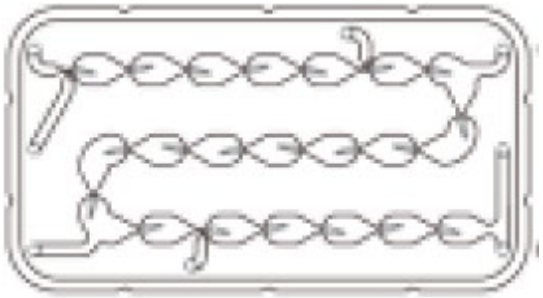
Rates/Phases	Homogeneous	Liquid-Liquid	Gas-Liquid	Solid-Liquid
Type A	Plate SZ/TG	Plate LL	Plate LL	CSTR/ Packed Bed
Type B	Plate SZ/TG Coil	Plate LL Coil pulsated	Plate LL Coil pressure	CSTR/ Packed Bed
Type C	Static mixer Coil	State mixer Coil pulsated	Static mixer Coil pressure	Coil pulsated



SZ (serpentine)



TG (tangential)



LL (liquid-liquid)

Plouffe, P.; Macchi, A .; Roberge, D. M. *Org. Proc. Res. Dev.,* **2014**, *18*, 1286-1294.

Answering the “What” of Flow

TRANSFORMERS AND GENERATORS

Transformers

Oxidation Modules
- O_2 via two-feed approach
- Tube in tube reactor
- Homogeneous oxidant & T-mixer

Reduction Modules
- H_2 via tube in tube combined with catalyst
- *In situ* H_2 generation—pressurized gas-liquid mixer & packed bed reactor
- Homogenous reductions (DIBALH)

Nitrations
- Mixed acid nitrations
- Nitration using acetyl nitrate (HNO_3-Ac_2O-AcOH)
- Alternative Nitration Condition (N_2O_5 + TFE; NH_4NO_3–H_2SO_4)

Olefinations
- Polymer-assisted HWE olefination using packed bed reactor
- Wittig reaction using SS coil at 210 ºC

Generators

In situ Hazardous Materials
- Stable Reagents
  - Cl_2, Br_2, BrCN, HCN, NOCl, N_2H_4, SO_2, $COCl_2$, $ClCH_2OCH_3$, R-NC
- Limited Stability Reagents
  - H_2O_2, RCO_3H, N_2CHCO_2Et, R-N_3, LDA
- Unstable Reagents
  - O_3, CH_2N_2, CF_3CHN_2, HN_3, BrN_3, ClN_3 $^1O_2$

Reactive Intermediates
- Iminium cations
- Benzyne
- R-MgX
- Aryllithium
- Carbon-centered radicals
- Alkyl diazo compounds
- Benzyl diazo compounds

Guidi, M.; Seeberger, P. H.; Gilmore, K. *Chem. Soc. Rev.*, **2020**, *49*, 8919-8932.
Dallinger, D.; Gutmann, B.; Kappe, C. O. *Acc. Chem. Res.*, **2020**, 53, 1330-1341.

Continuous Reactors for Energetic Materials Synthesis

Commercial-Off-The-Shelf Apparatus

LABORATORY REACTORS

Uniqsis FlowSyn

www.uniqsis.com

- All PTFE or PTFE–Hastelloy construction available
  - Temperature: –70 to +260 °C; Pressure: 100 bar
- Data Logger and FlowControl™ Software
  - Plan experiments; Monitor and log temperature, pressure, and flow rate
  - Wireless or remote control over LAN
- Up to 3 temperature zones and 3 reagent inputs available
- Available LED Photoreactor

Vapourtec RS-400



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- PFA or Hastelloy construction available
  - Temperature: –70 to +250 °C; Pressure: 50 bar
- Flow Commander™ Software
  - Plan experiments; Monitor and log temperature, pressure, and flow rate
  - API Package can be driven by .NET Software Framework
- Up to 4 reactors and 4 reagent inputs available
- Available Photoreactor (Hg lamp or LED) and Electrochemical reactor

Ehrfeld Modular MicroReaction System (MMRS)



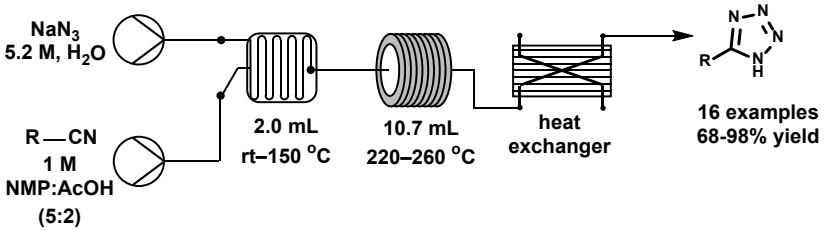
© Ehrfeld Mikrotechnik BTS GmbH.
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- Hastelloy construction available
  - Temperature: –20 to +200 °C (–100 to +600 °C); Pressure: 100 bar
- Integrates with LabManager® automation system from HiTec Zang
  - Consists of a control unit and LabVision® visualization and automation software
- More than 60 different microreaction modules
- Available Photoreactor (UV lamp or LED)

LABORATORY REACTORS

Uniqsis FlowSyn

www.uniqsis.com

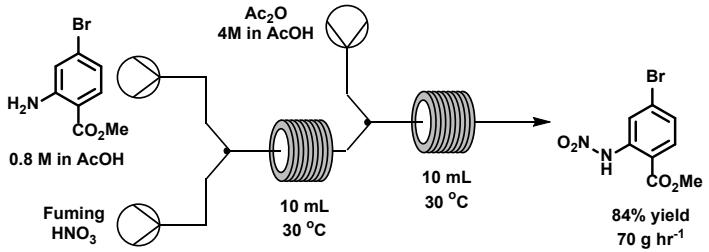


Gutman, B. et al. Angew. Chem. Int. Ed., **2010**, *49*, 7101-7105.

Vapourtec RS-400



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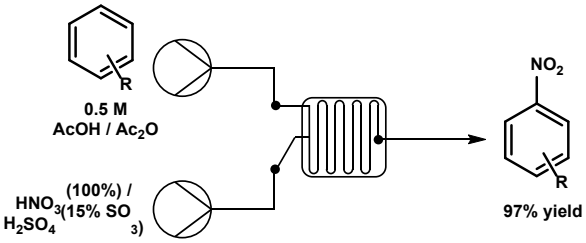


Brocklehurst, C. E.; Lehmann, H.; La Vecchia, L. *Org. Process Res. Dev.*, **2011**, *15*, 1447-1453.

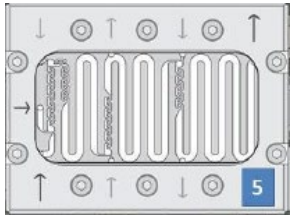
Ehrfeld Modular MicroReaction System (MMRS)



© Ehrfeld Mikrotechnik BTS GmbH. All rights reserved.



Köckinger, M. et al. *Org. Process Res. Dev.*, **2020**, *24*, 2217-2227.
Sagmeister, P. et al., *React. Chem. Eng.*, **2020**, *5*, 677-684.



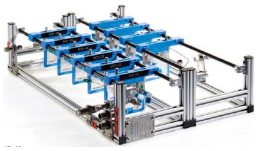
LL-Mixer 0.24 mL internal volume

SCALABLE REACTORS

Corning Advanced-Flow™ Reactors



© 2018 Corning Incorporated



© 2016 Corning Incorporated

- Borosilicate glass or SiC (G1 and larger) construction available
  - Temperature: –60 to +200 °C; Pressure: 18 barg
  - Metal-free system for high chemical durability
- Integrated thermostat; plug and play system with data monitoring
- Lab Photo Reactor Option
  - Tunable LED source with 6 different wavelengths; wireless control

Chemtrix Labtrix® S1 and Protrix®





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- Inert wetted materials:
  - Labtrix® S1: PTFE, ETFE, FFKM, Glass
  - Protrix®: PTFE, FFKM, SiC
  - Temperature: –20 to +195 °C; Pressure: 20 bar
  - Volume: 1 to 19.5 µL (Labtrix® S1); 1 to 13.5 mL (Protrix®)
- Dedicated software for automated data logging and sample collection
- Protrix® processes can be directly scaled to production (Plantrix®)

AM Technology Coflore® ACR





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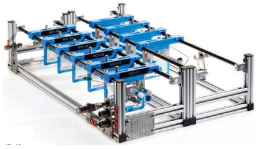
- Hastelloy reactor block; Hastelloy or ceramic agitators
  - Temperature: –40 to +140 °C; Pressure: 10 bar
- ACR cell block has 10 reaction cells connected via interstage channels
  - Sample points, addition points, & temp measurement can be added to any reaction cell
- Agitating platform provides horizontal agitation to reactor block for mixing
- Temperature data & agitation rates can be accessed by USB or LAN

SCALABLE REACTORS

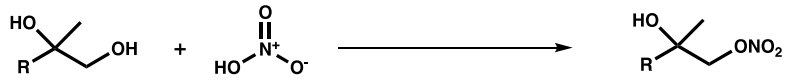
Corning Advanced-Flow™ Reactors



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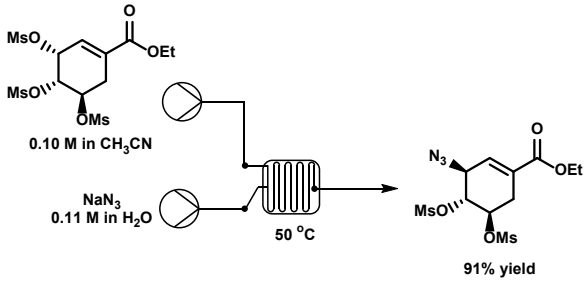
Braune, S. *et al. Chemistry Today*, **2008**, *26(5)*, 1-4.

Chemtrix Labtrix® S1 and Protrix®





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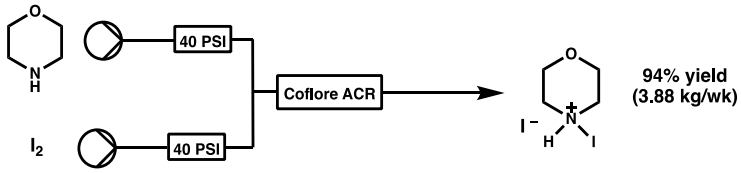
Sagandira, C. R.; Watts, P. *Beilstein J. Org. Chem.*, **2019**, *15*, 2577-2589.

AM Technology Coflore® ACR





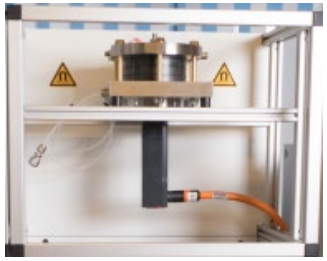
© AM Technology. All rights reserved.



Browne, D. L.; Deadman, B. J.; Ashe, R.; Baxendale, I. R.; Ley, S. V. *Org. Process Res. Dev.*, **2011**, *15*, 693-697.

SPINNING DISC REACTORS

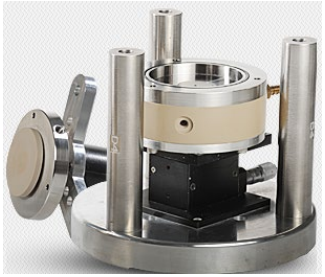
Flowid SpinPro R10



© Flowid BV - All rights reserved

- SiC Spinning Disc Reactor
  - Three-stage reactor; 19 mL total volume
  - Temperature: –20 to +160 °C; Pressure: 10 bar
- Discs can be mechanically or chemically modified
- Suitable for precipitations and for controlled emulsification
- Pilot (R300) and production (R1000) scale units available

KinetiChem Synthetron™

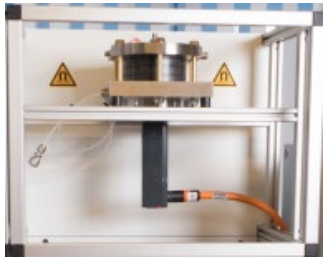


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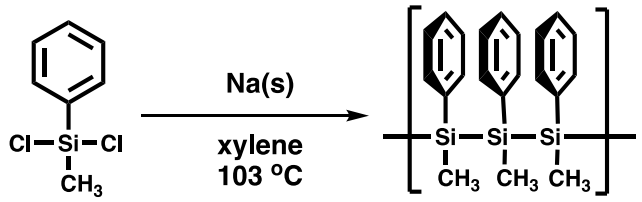
- Hastelloy C-22 surfaces; inert fluoropolymer seals
  - 10 µL to 1.3 mL lab scale spinning disc reactor
  - Temperature: –40 to +150 °C; Pressure: 10 bar
- 1,000 W motor; speed up to 14,000 RPM
- TouchScreen Allen Bradley Micro800 series controller/data logger
  - 4 x K-type thermocouple collection ports
  - 2 x 4-20 mA pressure transmitters

SPINNING DISC REACTORS

Flowid SpinPro R10

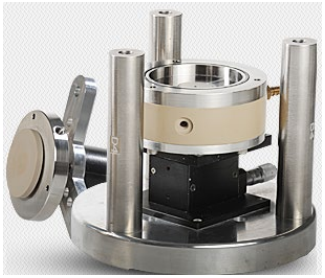


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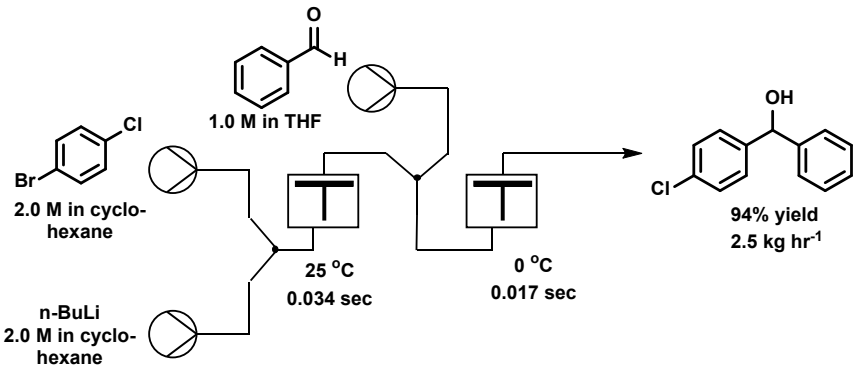


Hees, M.; Georgi, U.; Bachus, H.; Muller, K-S. US Patent Office US 2018/0346655, December 6, 2018

KinetiChem Synthetron™



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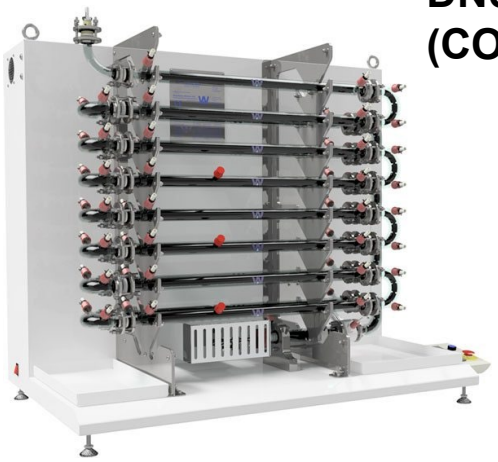
Slocum, D. W. *et al., Tetrahedron Lett.*, **2010**, *51*, 4793-4796.

NITECH® SOLUTIONS



DN25 Oscillatory Baffled Reactor (OBR)

- Borosilicate glass vessel; 25 mm diameter; 110 mL internal volume
- Temperature: –20 to +120 °C; Pressure: ambient
- Oscillator Frequency: 0.1 to 3 Hz in 0.1 Hz increments
- Oscillator Stroke: 5 to 40 mm in 1 mm increments
- May be configured for batch, semi continuous, or continuous operation
- Optimized OBR parameters may be used for COBC



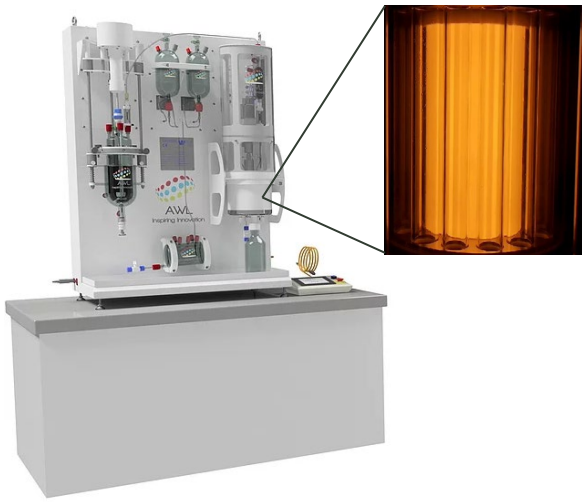
DN6/DN15 Continuous Oscillatory Baffled Crystallizer/Reactor (COBC/R)

- Glass (DN6/DN15) or Hastelloy (DN15); 200 mL (DN6)/1.25 L to 4.5 L (DN15) internal volume
- Temperature: 0 to +100 °C (DN6); –20 to +150 °C (DN15)
- Pressure: 0 to 3 bar (DN6); 0 to 10 bar (DN15)
- Oscillator Frequency: 0.1 to 6 Hz (DN6); 0.1 to 3 Hz (DN15)
- Oscillator Stroke: 2 to 25 mm (DN6); 11 to 68 mm (DN15)
- ATEX compliant DN15 models available

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Peña, R.; Olivia, J. A.; Burcham, C. L.; Jarmer, D. J.; Nagy, Z. K. *Cryst. Growth Des.*, **2017**, *17*, 4776-4784.
Kacker, R.; Maaβ, S.; Emmerich, J.; Kramer, H. AIchE J., **2018**, *64*, 2450-2461.

ALCONBURY WESTON CCF50 AND CCF20-LITE CONTINUOUS CAROUSEL FILTER



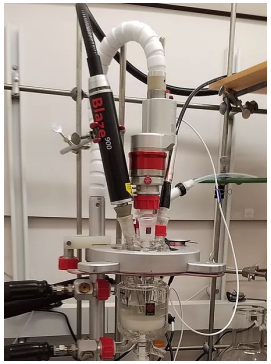
- **CCF50 For Hazardous Locations:**
  - Meets DOE Standard 1212 for Energetic Materials
  - No/Minimal particle shear
  - Up to three wash solvent reservoirs
  - 1 moving part
  - 50 mm ports – 40 g of material per port
  - Entry-to-exit time typically 10 to 15 minutes
  - Cake wetted to set parameters before discharge
  - Optional dryer
    - Dry gas fed to mass spec to monitor solvent content

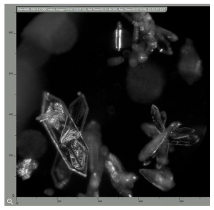


- **CCF20-Lite Features:**
  - New product provides CCF20 functionality w/o ancillaries
  - Auto transfer function from external reactor/crystallizer
  - Automated filtration and wash cycle
  - Optional N_2 blanket system
  - Optional cooling vessel for wash solvent

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BLAZE METRICS™ AND TORNADO SPECTRAL SYSTEMS





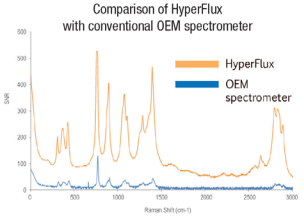
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One Probe with Multiple Integrated Technologies – Simultaneous Acquisition:

- **Microscopy:** high contrast, high resolution, highest dynamic range for understanding single dispersed phase and multiple phase, multiple component particle systems
- **High Dynamic Range Turbidity (HDR):** dynamic range measuring from low to extremely high dispersed phase concentrations; can track change at nano or micron scale, measure optical transitions in liquid and/or solid phase, operate in translucent to black solutions
- **Advanced Chord Length (A-CLD):** removes flow speed artifacts and reduces multiple other artifacts of scanning tools; track changes in particle size, count, and shape
- **Particle Focused Raman (PFR):** can dramatically increase Raman signal captured from dispersed phase particles; find and track polymorphs, solvates, hydrates, impurities; differentiate multiple component systems.
- **Immersed Probe Tip Material:** Hastelloy 22, 276, SS, or custom
- **Window Materials:** Sapphire, Kalrez, Nickel, and Gold plate
- **Temp:** -10 to 100 ^{o}C (-10 ^{o}C requires N_2 purge)
- **Pressure:** 6 bar (standard); 22 bar (optional)

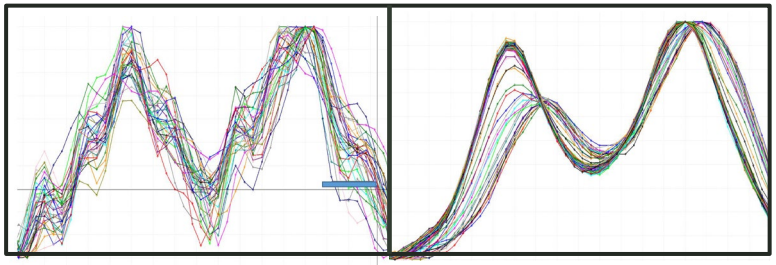
- **HyperFlux™ Pro Plus Raman**
  - 200–3300 cm^{-1} optical range
  - Laser Power: 20 mW to 495 mW (ATEX compliant)
  - Excitation wavelength: 785 nm





Ball Probe w/Tornado (pre-processed)

Blaze PFR Probe w/Tornado (raw data)



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MAGRITEK BENCHTOP NMR



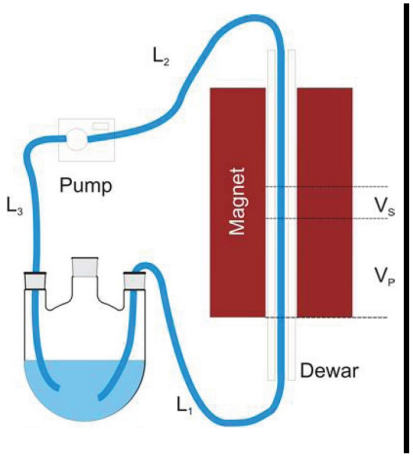
- **Specifications**
  - 43, 60, or 80 MHz (^{1}H)
  - 23” x 17” x 16”
  - 120 lbs
  - 110–240 AC, 60 Hz
  - Operating Temp: 20 to 25 ^{o}C
  - 2 G line completely inside spectrometer

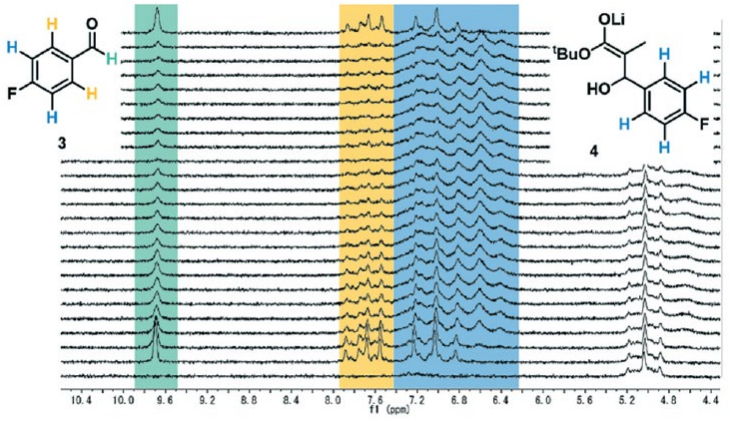
- **Available Pulse Sequences**

- Proton
  - 1D
  - Paramagnetic
  - 2D COSY
  - 2D TOCSY
  - 2D JRES
  - T_1, T_2
  - Reaction Monitoring

- Carbon
  - 1D
  - DEPT
  - HETCOR
  - HMBC
  - HMQC
  - HSQC
  - HSQC-ME

- Optional third nuclei: ^{7}Li, ^{11}B, ^{15}N, ^{23}Na, ^{29}Si, ^{19}F, and ^{31}P





Sagmeister, P.; Willimas, J. D.; Hone, C. A.; Kappe, C. O. *React. Chem. Eng.*, **2019**, *DOI: 10.1039/c9re00087a*

PUMPS

Vapourtec SF-10



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- Self-priming
- Flow rate: 0.02 to 10 mL min^{-1}
- Max pressure: 10 bar
- Pumps solutions, suspensions, light slurries, and gasses
- Versatile options for external control

- Operating Modes:
  - Constant flow rate or ramped flow rate
  - Volume dosing
  - Gas delivery
  - Pressure controller

Fuji Techno SMP

www.fuji-techno.co.jp/english

- Super Metering Pump is pulse-free; metal-free version available
- Able to feed liquid at ± 0.1% of specified flow range
- Max flow rate: 15.3 to 108.6 mL min^{-1}
- Max discharge pressure: 20 bar

- Applications
  - Additive feed into extruder
  - High precision dosing
  - Line mixing
  - Emulsification

Teledyne ISCO



© Teledyne ISCO. All rights reserved.

- Seven D-series pumps
  - Flow rates: < 1 µl min^{-1} to 400 mL min^{-1}
  - Max pressure: > 2000 bar
  - HLf-series pumps conform to UL Class I, Div 2, Groups A, B,C, & D, T4 environments
- Air and electric valve continuous flow systems
  - Max Temp: 160 °C (air); 200 °C (electric)
  - Flow rates: 1 µl min^{-1} to 133 mL min^{-1}
  - Max Pressure: > 1300 bar

SEPARATORS

Zaiput Flow Technologies



Liquid–Liquid/Gas–Liquid Separators

- Provide continuous separation of immiscible phases by exploiting differences in wettability of a porous membrane
- Max Temperature: 130 °C; Max Pressure: 20 bar
- Wetted parts: ETFE, PFA, FEP, PTFE
- 0.5 mL internal volume; 0 – 10 ml min^{-1} total flow rate

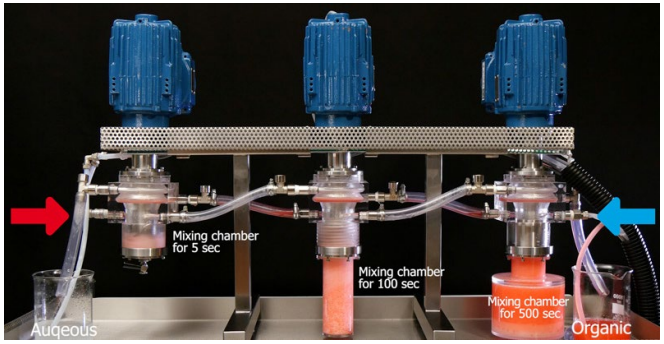


Multi-Stage Extraction Platform

- Bench-scale tool for countercurrent liquid-liquid extraction
- Max Temperature: 80 °C
- Wetted parts: ETFE, PFA, FEP, PTFE, FFMK, PVDF
- ~3 mL per stage internal volume, 0 – 10 ml min^{-1} total flow rate

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CINC Centrifugal Extractors



- Continuous mixing / extraction / separation in one step
- Device can be retrofitted to increase the mixing time in a larger mixing volume
- Lab scale results easily scaled up to production
- Hastelloy C22 construction available; ATEX compliant
- Temperature –30 to +130 °C; Pressure: 20 bar
- 0 – 1000 ml min^{-1} total flow rate

© CINC Deutschland GmbH & Co. KG

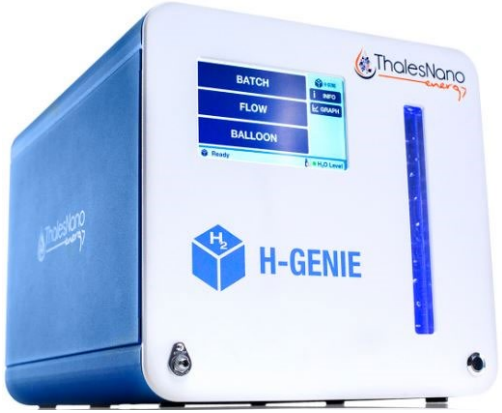
HYDROGEN GAS GENERATORS



© ThalesNano, Inc. All rights reserved.

ThalesNano H-Cube™ Series

- Hydrogenation without cylinders
- Electrolytic cells generate H_2 up to 60 NmL/min and 100 bar
- Temperature range: 10 to 150 °C
- Flow rates: 0.3 to 3 mL/min



© ThalesNano, Inc. All rights reserved.

ThalesNano H-Genie™ High Pressure H_2 Generator

- Generates ≥ 99.99% purity H_2
- Gas flow range: 0.1 to 1 NL
- Pressure range: 1 to 100 bar
- Temperature range: 10 to 150 °C
- Suitable for batch, continuous, and balloon fill operation
- Monitors and records H_2 consumption
- Uses < 1 μS cm^{-1} water
- Requires ion filter for H_2O reservoir (consumable)

SUMMARY AND ADDITIONAL RESOURCES

- **Advantages of flow chemistry**
  - Safety
  - Expansion of reaction space to include “forbidden” reactions
  - Lower CAPEX and OPEX
  - Reduced footprint
  - Ease of scale-up

- **Advances in continuous reactor technology have expanded the equipment capability envelope to accommodate an increasing number of process demands**

Additional Resources

- *Flow Chemistry*, Volumes 1 & 2 by F. Darvas, G. Dormán, and V. Hessel (Eds.)
- *The Hitchhikers Guide to Flow Chemistry, Chem. Rev.*, **2017**, *117*, 11796–11893.
- Flow Chemistry and Continuous Processing Conference (virtual) 10 – 12 May, 2021
- Flow Chemistry Congress 2021, Boston, MA (Dates to be confirmed)
- **Questions?**
  - joseph.rheinhardt@csssquaredllc.com

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  - Dr. Kimberly Spangler, Program Manager, Joint DoD/DOE Munitions Program OUSD (R&E)
  - Dr. Paritosh Dave, Senior Program Manager, Leidos







U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Quantitative Structure-Activity/Property Relationship Models for Secondary Explosive Compounds

Carson Britt

Research Scientist I

CS Squared LLC

Approved For Public Release

BOTTOM LINE UP FRONT

- **Developed models to rapidly predict the properties of secondary explosive compounds from molecular structure alone**
  - Quantitative structure-activity/property relationships (QSAR/QSPRs) use regression or classification algorithms to relate structural features to properties
  - Density, melting temperature, vapor pressure, impact sensitivity, oral rat LD50 (LD50), 48 hour *Daphnia magna* LC50 (LC50DM), 96 hour fathead minnow LC50 (LC50FM), 40 hour *Tetrahymena pyriformis* IGC50 (IGC50), and bioaccumulation factor (BCF)

- **Statistically validated QSAR/QSPR's for accuracy, applicability domain**
  - Accuracy quantified by (repeated) nested five-fold cross validation
    - Root mean square error, mean absolute error, median error, Pearson correlation coefficient
  - Applicability domain by Tanimoto similarity to training set, predicted value
    - Gauge reliability of property predictions for new and unknown compounds

- **LiveDesign platform hosts QSAR/QSPR models, other tools**
  - Models available in easy to use, online interface

SCOPE AND AGENDA

- **Objective: using a dataset of "ground truth" data, establish correlations between the molecular structure and physical properties of known compounds. Use these correlations to predict the physical properties of new compounds from only their molecular structure.**

- **Requirements for a QSAR/QSPR model:**
  - Dataset of molecular structures and their properties
  - Routine to represent molecular structures in a machine understandable way
  - Algorithm to elucidate correlations between molecular representations and the corresponding physical properties
  - Criteria to evaluate the accuracy of correlations when applied to new compounds

- **Agenda:**
  - Background machine learning & QSAR/QSPR concepts and terminology
  - Technical aspects of QSAR/QSPR model development
  - Evaluation of accuracy and applicability of the developed models
  - Use of the models in new compound discovery workflow

Quantitative Structure-Activity/Property Relationship Models for Secondary Explosive Compounds

Fundamental machine learning & QSAR/QSPR concepts and terminology

TYPES OF MODELS

- **Minimizing error with supervised regression models**
  - Model selection – Linear, logistic, k-nearest neighbors, kernel ridge regression (KRR), support vector machines (SVM), decision trees, neural networks
  - Regularization – L1 regularization, L2 regularization, dropout
  - Model ensembling – combining multiple models to reduce bias and variance

- **For small structured datasets ensemble decision tree models are consistently among the best performing models**
  - Exhibit excellent predictivity
  - High level of interpretability
  - Relatively computationally inexpensive
  - Hyperparameters easily adjusted to needs of a given dataset

- **Gradient boosting algorithms**
  - CatBoost
  - LightGBM
  - XGBoost

GRADIENT BOOSTING

Input Dataset



Train decision tree



Predict on training data
Calculate residual prediction error
Use residual error as new training input

Train decision tree



Predict on training data
Calculate residual prediction error
Use residual error as new training input

Train decision tree



Predict on training data
Calculate residual prediction error
Use residual error as new training input

Sum outputs of each tree

Prediction

Quantitative Structure-Activity/Property Relationship Models for Secondary Explosive Compounds

Model Development

QSAR/QSPR MODEL DEVELOPMENT

- **A QSAR/QSPR model needs:**
  - Dataset of molecular structures and their physical properties
    - Molecules represented via simplified molecular-input line-entry system (SMILES)
  - Routine to represent molecular structures in a machine understandable way
    - RDKit – cheminformatics python library
  - Algorithm to elucidate correlations between molecular representations and the corresponding physical properties
    - XGBoost, dask, scikit-learn, hyperopt – machine learning python libraries
  - Criteria to evaluate the accuracy of correlations when applied to new compounds
    - scikit-learn, SciPy, NumPy, pandas – math and data visualization python libraries

DATASETS

Physical Property	# Compounds	Type Compounds	Sources
Density	15,435	Small molecules, drug-likes, energetics	ochem.eu, EMD
Melting temperature	3,171	Small molecules, drug-likes, energetics	DPG, EMD
Vapor pressure	3,268	Small molecules, pesticides, drug-likes, energetics	ochem.eu, EMD
Impact sensitivity	308	Energetics	Didier Mathieu
Oral rat LD50	7,294	Small molecules, pesticides, drug-likes	EPA T.E.S.T.
48 hour *Daphnia magna* LC50	353	Small molecules, pesticides, drug-likes	EPA T.E.S.T.
96 hour fathead minnow LC50	823	Small molecules, pesticides, drug-likes	EPA T.E.S.T.
40 hour *Tetrahymena pyriformis* IGC50	1,792	Small molecules, pesticides, drug-likes	EPA T.E.S.T.
Bioaccumulation factor	672	Small molecules, pesticides, drug-likes	EPA T.E.S.T.

DATASETS

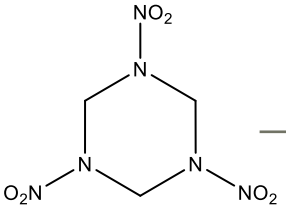
- **Sources of data**
  - Didier Mathieu impact sensitivity dataset (Didier Mathieu)
    - Mathieu, D., "Sensitivity of Energetic Materials: Theoretical Relationships to Detonation Performance and Molecular Structure“, Ind. Eng. Chem. Res., vol 56, no. 29, 2017, pp. 8191-8201
  - Jean-Claude Bradley double plus good melting point dataset (DPG)
    - Bradley, J-C., Lang, A., Williams, A.J., “Jean-Claude Bradley double plus good (highly curated and validated) melting point dataset”, 2019.
  - Energetic Materials Database (EMD)
    - Britt, C., and Hrudka, J., “Energetic Materials Database”, CS Squared LLC, 2019.
  - EPA Toxicology Estimation Software Tool (EPA T.E.S.T.)
    - Martin, T., “User’s Guide for T.E.S.T. (Toxicity Estimation Software Tool), U.S EPA/National Risk Management Research”, 2016.
  - Online Chemical Modeling Environment (ochem.eu)
    - Tetko, V. I., *et al*., “Online chemical modeling environment (OCHEM): web platform for data storage, model development and publishing of chemical information”, J. Comput. Aided Mol. Des., vol. 25, no. 6, 2011, pp. 533-554
- **Datasets curated prior to use in model building**
  - No duplicates, salts, charged species, mixtures
  - No compounds containing elements besides C, H, N, O, B, P, S, F, Cl, Br, and I

NUMERICALLY REPRESENTING MOLECULES

- **Sample rows from melting point dataset:**

Compound	SMILES String	Melting Point (°C)
HMX	[O-][N+](=O)N1CN(CN(CN(C1)[N+](=O)[O-])[N+](=O)[O-])[N+](=O)[O-]	275
RDX	[O-][N+](=O)N1CN(CN(C1)[N+](=O)[O-])[N+](=O)[O-]	204
LLM-200	Nc1nonc1/N=[N+](\c1nonc1N)/O	182
TNT	[O-][N+](=O)c1cc([N+](=O)[O-])c(c(c1)[N+](=O)[O-])C	81
BODN	O=[N+](OCC1=NC(C2=NOC(CO[N+]([O-])=O)=N2)=NO1)[O-]	82

  - Convert SMILES to numeric format that can be used by XGBoost



CC1=C([N+]([O-])=O)C=C([N+]([O-])=O)C=C1[N+]([O-])=O

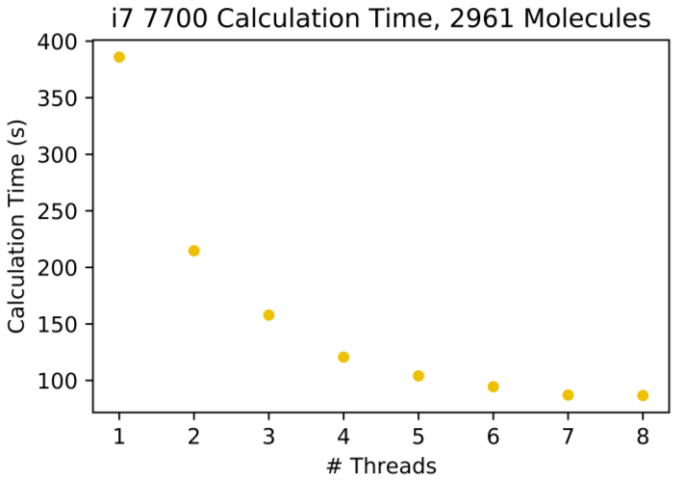
Atom counts
Functional group counts
Estate fingerprint
Etc.

- **Use RDKit to calculate 1447 molecular features**
  - Zero dimensional: Atom counts, atom ratios, oxygen balance, etc.
  - One dimensional: Bond counts, bond ratios, information indices, etc.
  - Two dimensional: Functional group fragments, fingerprints, etc.
  - Three dimensional: WHIM, geometric distances, inertial, etc.
- **Property and molecular structure now represented numerically:**

[275, 1.959, 0.476, 0.4, 0, … 12.453]

MOLECULAR FEATURE CALCULATION & FEATURE SELECTION

- **Calculation of molecular features not computationally expensive**
  - RDKit python library well optimized for an interpreted language
  - Calculation of molecular features is a trivially parallelized task



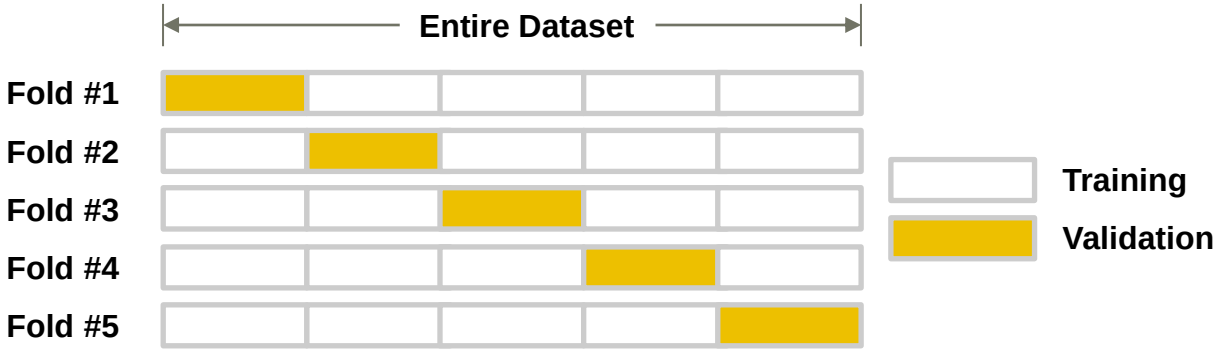
Number Threads	Calculation time: Intel i7 7700 (s)	Calculation time: AMD 2950x (s)
1	386	496
2	215	254
4	121	140
8	87	73
16	N/A	48
32	N/A	42

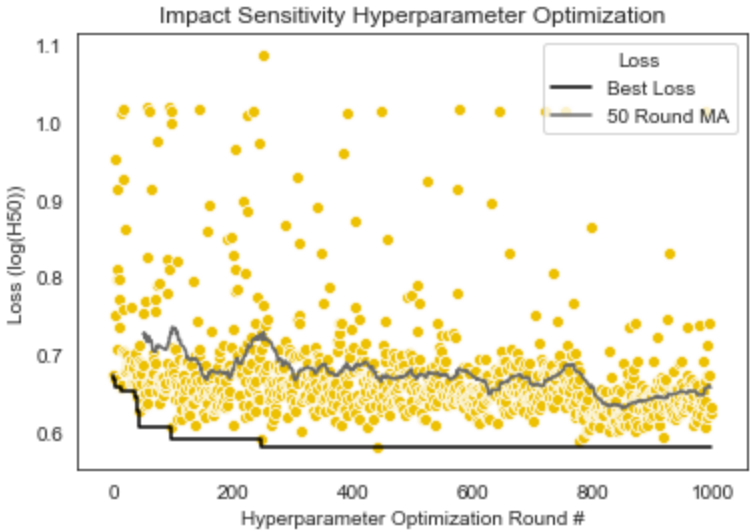
- **Feature selection using Boruta method**
  - Feature selection can improve model accuracy, interpretability, decrease wall-time
  - Duplicate each feature, y-scramble the rows creating “shadow features”
  - Use shadow features and original features to train a random forest model
  - Rank feature importance within random forest model, reject original features that perform worse than specified percentage of shadow features
  - Features that perform better than random noise are retained

Kursa, B. M., Jankowski, A., Rudnicki, W. R., “Boruta – A system for feature selection” Fundamenta Informaticae, vol. 101, 2010, pp. 271–285, doi: 10.3233/FI-2010-288

HYPERPARAMETER OPTIMIZATION

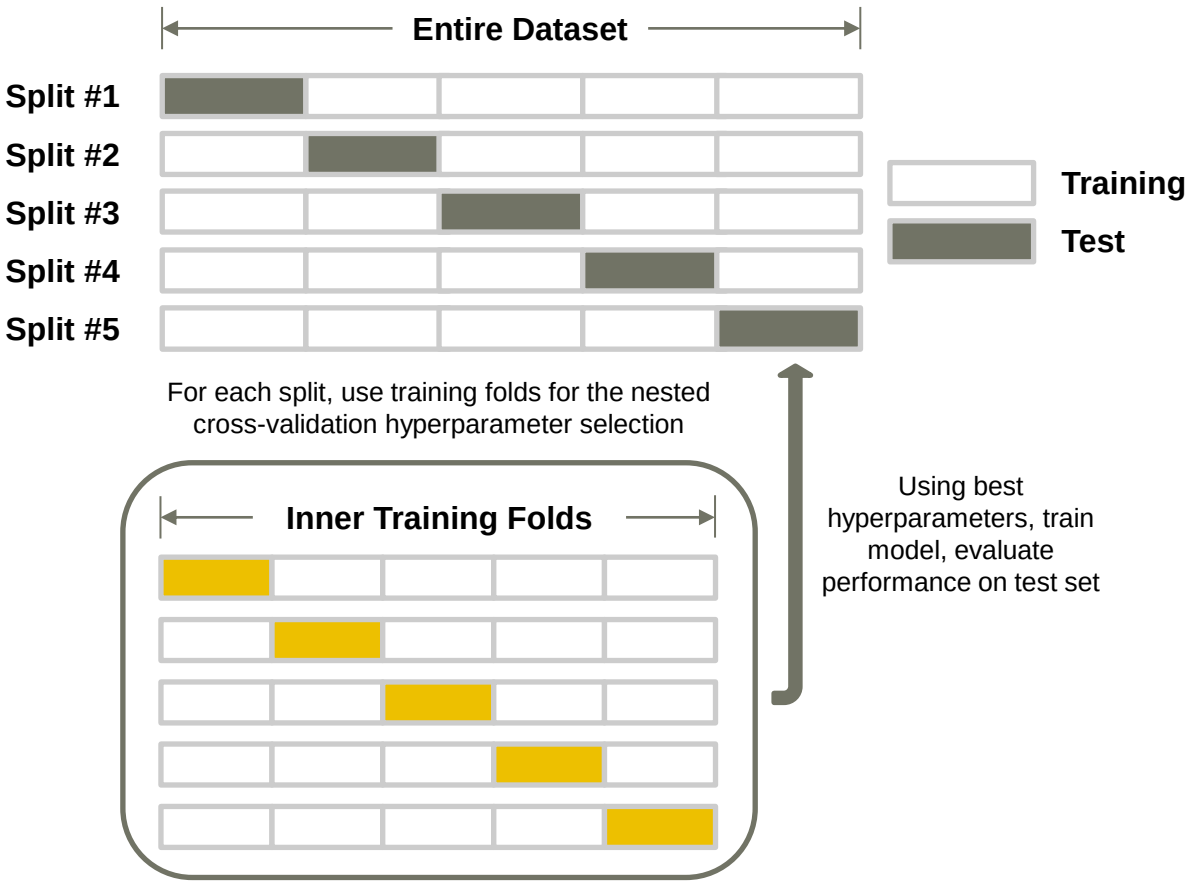
- **Bayesian hyperparameter tuning**
  - Hyperparameters are specifications for how a model is constructed, i.e., number of boosting rounds, max depth, etc.
  - Generate distribution of possible hyperparameters
  - Test a set of hyperparameters with cross-validation, use test results to inform the selection of the next set of hyperparameters to try
  - Converge toward optimal set of hyperparameters





Bergestra, J., Yamins, D., Cox, D. D., "Making a science of model search: hyperparameter optimization in hundreds of dimensions for vision architectures" ICML'13: Proceedings of the 30th International Conference on International Conference on Machine Learning, vol. 28, 2013, pp. 115-123

NESTED K-FOLD CROSS VALIDATION



- **Algorithm:**
  - Divide data into k folds
  - Withhold each fold once as a test fold, use remaining folds for feature selection, hyperparameter selection, and training
  - Divide training folds again into k equal inner training folds, select features
  - Hyperparameters selected with cross validation
  - Use hyperparameters to train model with all inner training folds
  - Evaluate model on the withheld test set
  - Repeat for each split
- **Repeat algorithm**

Quantitative Structure-Activity/Property Relationship Models for Secondary Explosive Compounds

Model Evaluation

QSAR/QSPR STATISTICAL PROPERTIES

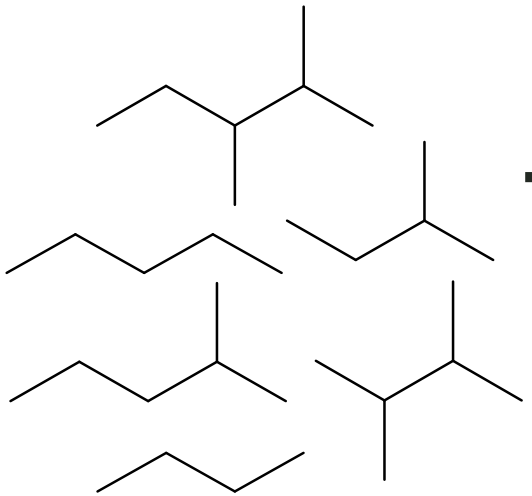
Property	MAE	RMSE	Median	Q-squared
Density (g/cm³)*	0.0214	0.0364	0.0119	0.988
Melting temperature (°C)*	25.5	34.68	19.34	0.873
Vapor pressure (Log10(mmg))*	0.531	0.874	0.271	0.942
Impact sensitivity (ln(H50))	0.458	0.616	0.335	0.629
LD50 (-Log10(mol/kg))*	0.420	0.573	0.317	0.638
LC50DM (-Log10(mol/L))	0.795	1.065	0.594	0.618
FC50FM (-Log10(mol/L))	0.593	0.827	0.410	0.692
IGC50 (-Log10(mol/L))	0.321	0.455	0.223	0.812
BCF (Log10)	0.466	0.625	0.342	0.791

* Results from only one round of external CV

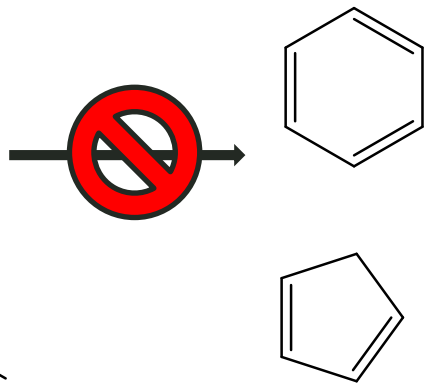
APPLICABILITY DOMAIN

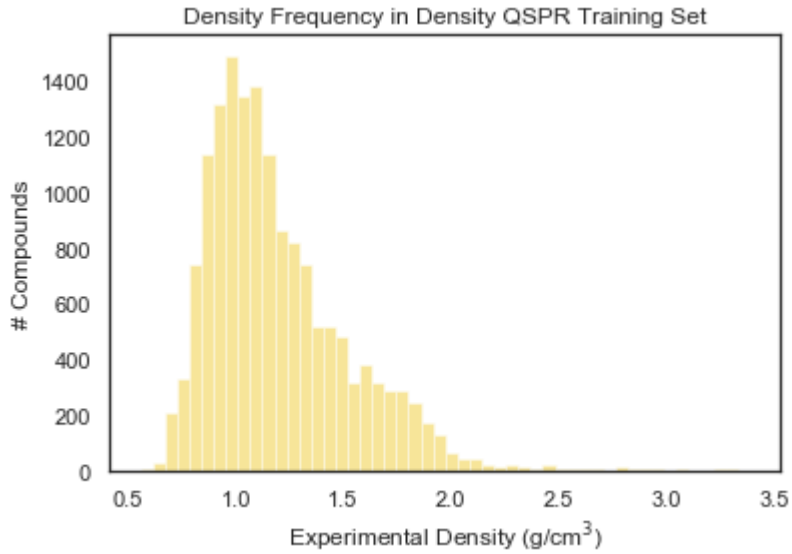
- **Physical properties of some molecules will be better predicted than others, quantify confidence in prediction for specific molecule**
  - Applicability domain is the chemical space where models will give good predictions
  - Predictions for molecules that are similar to training set molecules will be better
  - Numeric value of predicted property can also be used to gauge prediction accuracy

Training set

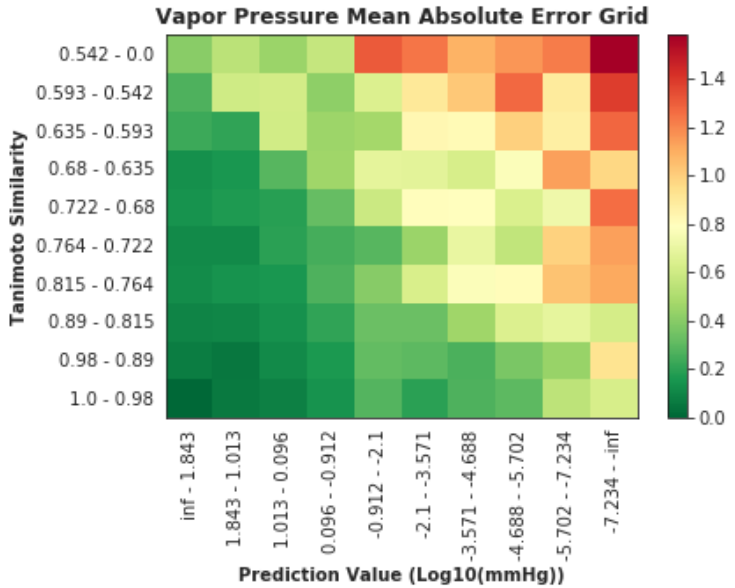


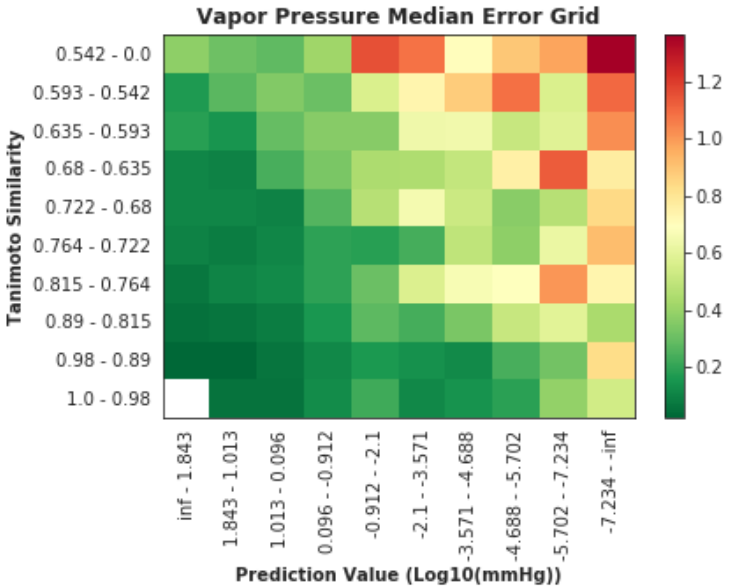
Test set

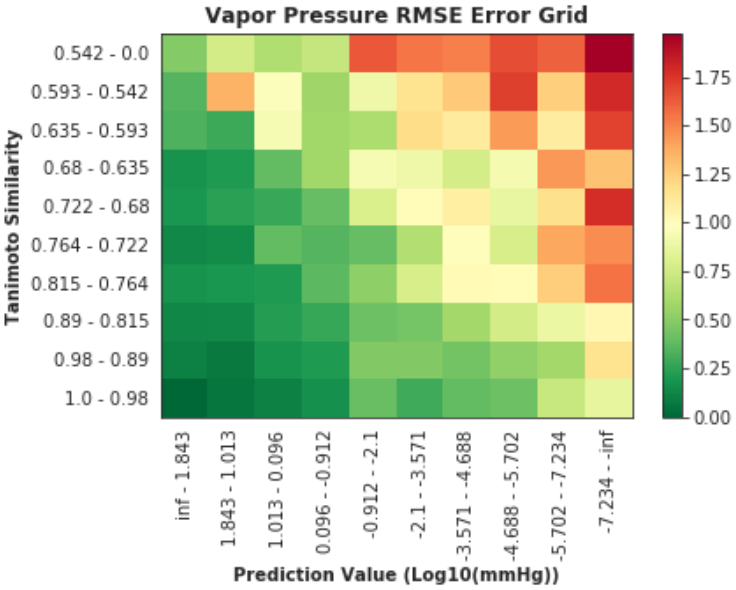


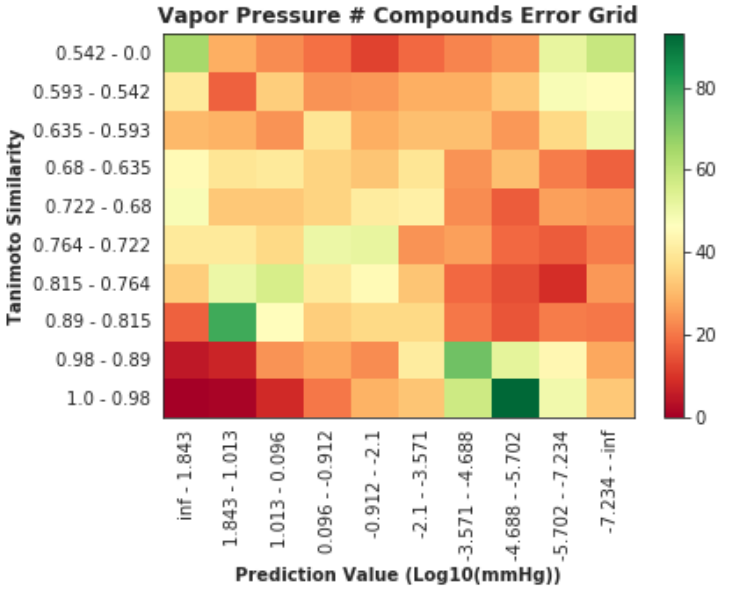


APPLICABILITY DOMAIN







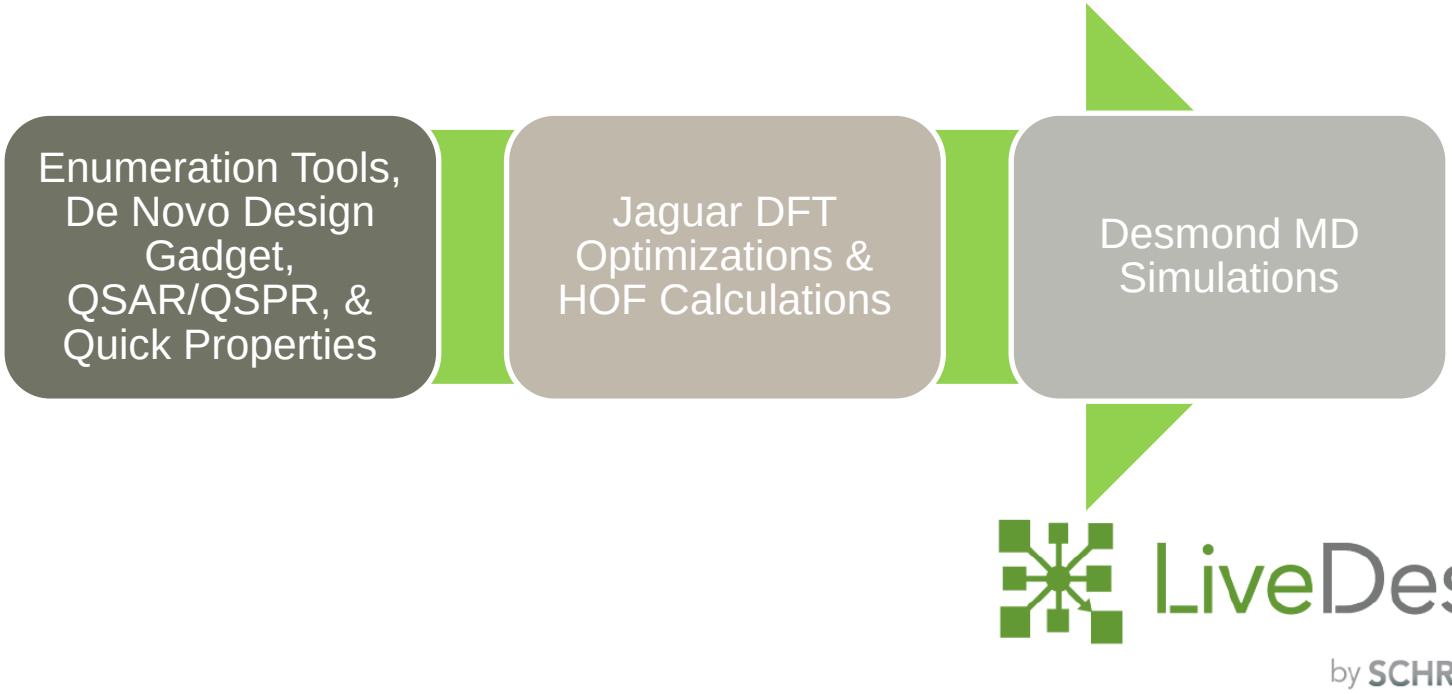


Quantitative Structure-Activity/Property Relationship Models for Secondary Explosive Compounds

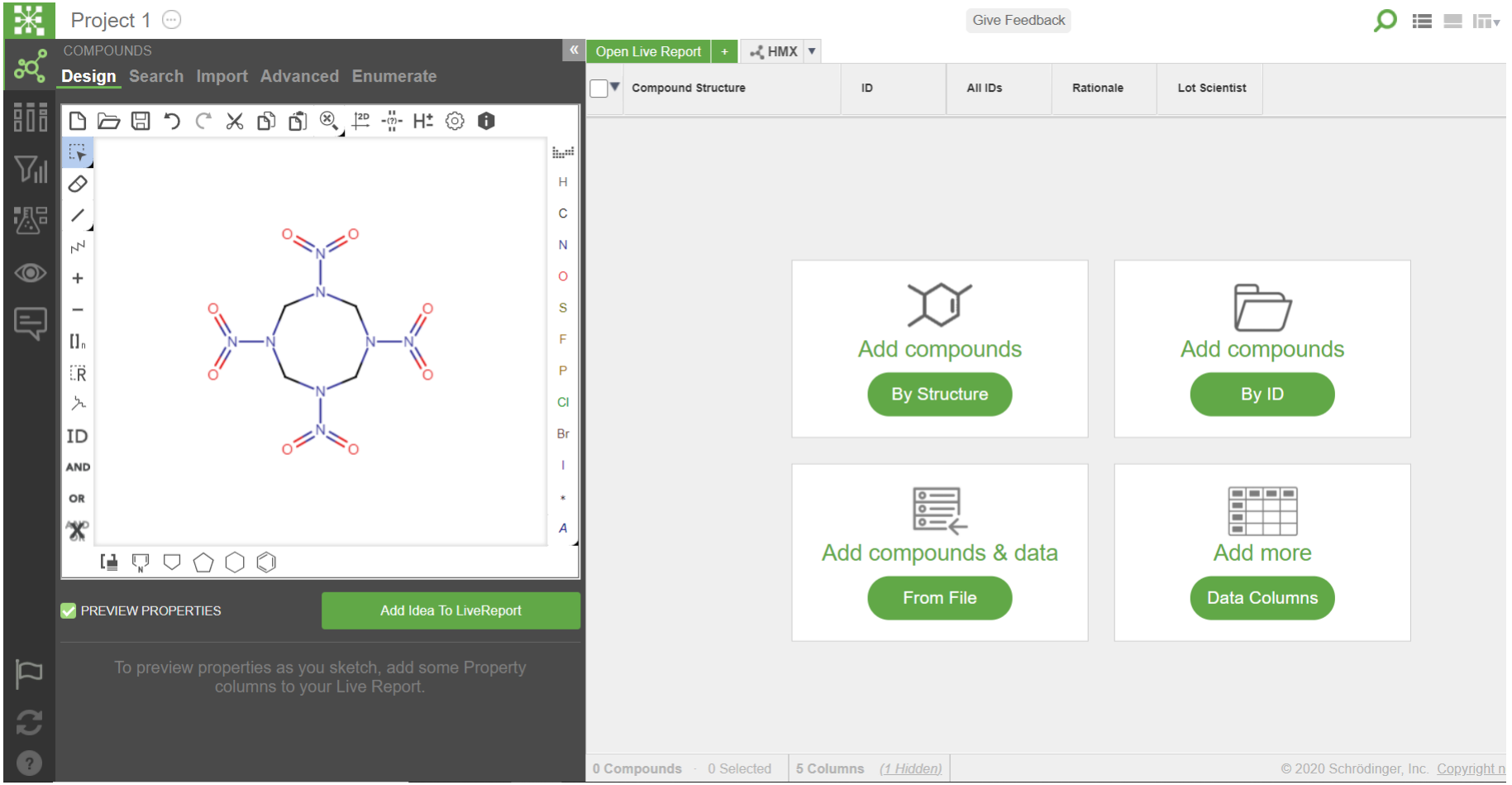
Implementation in LiveDesign

LIVEDESIGN: WEB-BASED COLLABORATION

- **LiveDesign by Schrödinger**
  - An online informatics platform that allows teams to collaborate, design, and experiment in one centralized location
  - Enables teams of computational and synthetic chemists and engineers to work together and share results on one platform
  - Add data, visualize structures, run calculations with a single click
  - Keeps data and files in one place that can be easily searched and accessed



LIVEDESIGN: ADDING COMPOUNDS



LIVEDESIGN: MAKING PREDICTIONS

Project 1

Give Feedback

DATA & COLUMNS

Project LiveReport

Search columns

- Density
- Melting Point
- Vapor Pressure
  - Mean Prediction Error
  - Median Prediction Error
  - Prediction
- Toxicology Models
  - BCF
    - Mean Prediction Error
    - Median Prediction Error
    - Prediction
  - IGC50
  - LC50DM
  - LC50FM
  - LD50
    - Mean Prediction Error
    - Median Prediction Error
    - Prediction

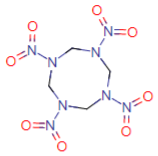
Experimental Assays

Other Columns

f(x) Formulas NEW

Add Columns

Open Live Report + HMX

	Compound Structure	ID	Vapor Pressure (Prediction)	BCF (Prediction) [log]	LD50 (Prediction) [-log(mol/kg)]
1		V35008	-13.1	0.6	1.8

1 Compounds · 0 Selected | 5 Columns (4 Hidden)

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RECAP

- **Developed models to predict the properties of secondary explosive compounds from molecular structure alone**
  - Fast molecular descriptor calculation, feature selection with Boruta method
  - Bayesian hyperparameter optimization
  - Models trained in parallel using GPU's
  - Models developed for density, melting temperature, vapor pressure, impact sensitivity, IGC50, LD50, LC50DM, LC50FM, and BCF
- **Statistically validated QSAR/QSPR's for accuracy, applicability domain**
  - Accuracy quantified by (repeated) nested five-fold cross validation
    - Root mean square error, mean absolute error, median error, Pearson correlation coefficient
  - Applicability domain by Tanimoto similarity to training set, predicted value
    - Gauge reliability of property predictions for new and unknown compounds
- **LiveDesign platform hosts QSAR/QSPR models, other tools**
  - Models available in easy to use, online interface
- **Questions?**
  - carson.britt@csssquaredllc.com

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MSIAC
Munitions Safety Information Analysis Center

Supporting Munitions Safety



Risk Associated with Under Classification of Small Arms Ammunition

IMEMTS 2021

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Introduction

- Conventional small arms ammunition (SAA) below .50 Cal. (12.7 mm) has traditionally been classified as 1.4 S.
- As a reminder, the UN classification divides Dangerous Goods Class 1 materials in 6 subdivisions (1.1 to 1.6) and 13 compatibility groups (CG).
  - **Division 1.4** = Substances and articles which present **no significant hazard**
  - **Compatibility Group S** = Substance or article so packed or designed that **any hazardous effects arising from accidental functioning are confined within the package** unless the package has been degraded by fire, in which case all blast or projection effects are limited to the extent that they do not significantly hinder or prohibit fire fighting or other emergency response efforts in the immediate vicinity of the package



Introduction

- Question: is this classification always appropriate? In any situation?

- Where SAA is under classified, this may result in an **underestimation** of the actual risk posed by the ammunition.

- In order to get a better overview on how the nations classify their munitions, MSIAC has conducted an **international review** in the 2016-2020 timeframe, with the help of MSIAC and NATO contributing nations and the national HC databases hosted by MSIAC (see the MSIAC limited report L-215 for more detailed information).

- This presentation provides the output of this work with a focus on small arms ammunition (SAA) of .50 Cal. (12.7 mm) or less.

I. Hazard Classification in the Nations

I. Hazard Classification in the Nations

- MSIAC hosts a Hazard Classification database with input from 16 nations
- Data has recently been compared and analysed (L-215)

Table 10 : Available Information in most recent national contributions to the MSIAC HC database

	Australia	Belgium	Canada	Czech Rep.	Finland	France	Germany	Latvia
Type of file	Access	Access	Access	PDF	Excel	Excel	Excel	Excel
Number of articles (Total)	7412	1066	2479	207	282	3120	2619	357
Number of articles from class 1	7412	1066	2479	207	282	3120	2619	357
Most recent contribution (year)	2019	2019	2019	2019	2019	2019	2019	2019
Hazard Storage sub-Division	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Compatibility group	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Sensitivity group	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Rejected from Class 1	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
1.1	7412	1066	2479	207	282	3120	2619	357
1.2	7412	1066	2479	207	282	3120	2619	357
1.2.1 (SsD)								
1.2.2 (SsD)								
1.2.3 (SsD)								
1.3	7412	1066	2479	207	282	3120	2619	357
1.3.1 (SsD)								
1.3.2 (SsD)								
1.4	7412	1066	2479	207	282	3120	2619	357
1.5	7412	1066	2479	207	282	3120	2619	357
1.6	7412	1066	2479	207	282	3120	2619	357
NSN	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
UN Number	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
NEQ	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
MCE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Packaging	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

	Netherlands	Norway	Poland	Slovakia	Slovenia	Ukraine	UK	US
Type of file	Excel	Excel	Access	Excel	Excel	Excel	Excel	Excel
Number of articles (Total)	1357	1221	593	484	337	214	3855	18175
Number of articles from class 1	1355	885	594	463	337	202	3852	17013
Most recent contribution (year)	2012	2009	2011	2010	2018	2008	2018	2019
Hazard Storage sub-Division	No	Yes	No	No	Yes	No	Yes	Yes
Compatibility group	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Sensitivity group	No	No	No	No	Yes	No	No	Yes
Rejected from Class 1	No	No	No	Yes	No	No	Yes	Yes
1.1	364	269	195	121	99	***	969	4786
1.2	168	155	126	77	20	***	584	2670
1.2.1 (SsD)		(36)			(5)		(125)	(1473)
1.2.2 (SsD)		(119)			(12)		(428)	(1138)
1.2.3 (SsD)		(0)			(0)		(4)	(59)
1.3	326	129	62	81	27	***	671	2876
1.3.1 (SsD)		(29)			(8)		(159)	
1.3.2 (SsD)		(99)			(19)		(468)	
1.4	515	332	210	121	191	***	1628	7101
1.5	0	0	0	0	0	0	0	0
1.6	0	0	0	0	0	0	0	0
NSN	Yes	Yes	No	Yes	Yes	No	No	Yes
UN Number	Yes	No	Yes	Yes	Yes	No	Yes	Yes
NEQ	Yes	Yes	Yes	Yes	No	Yes	Yes	Yes
MCE	No	No	No	No	No	No	No	Yes
Packaging	Yes[6]	Yes	No	Yes	No	No	Yes	Yes[7]



L-215
April 2020

A REVIEW OF INTERNATIONAL HAZARD CLASSIFICATION

Martijn VAN DER VOORT
Christelle COLLET
Matthew FERRAN

MSIAC UNCLASSIFIED

I. Hazard Classification in the Nations

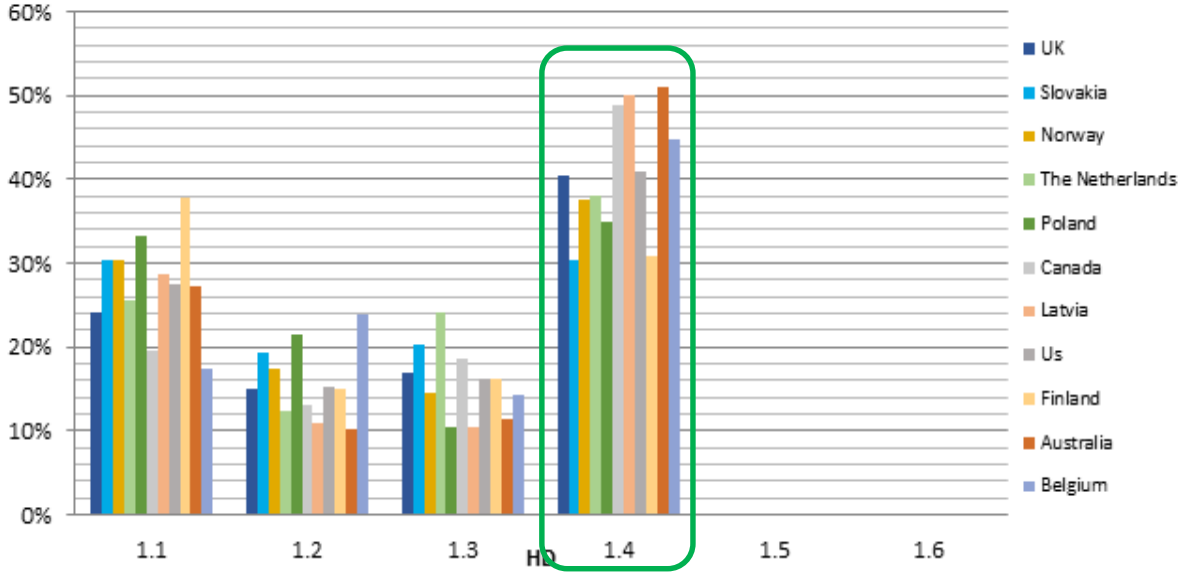
- Large differences in provided data format and fields.
  - Use of Storage sub Divisions
  - Use of Sensitivity Groups
  - Maximum Credible Event (relevant for HD1.2)
  - NSN, packaging information

- Development of an automated HC Database Exchange (HCDx).
  - Search across all nations and data fields for hazard classification data
  - Specifications are ready, development planned for 2022

I. Hazard Classification in the Nations

- Items in Hazard Division 1.4 often represent the highest percentage of HD assignments in the contributing nations.

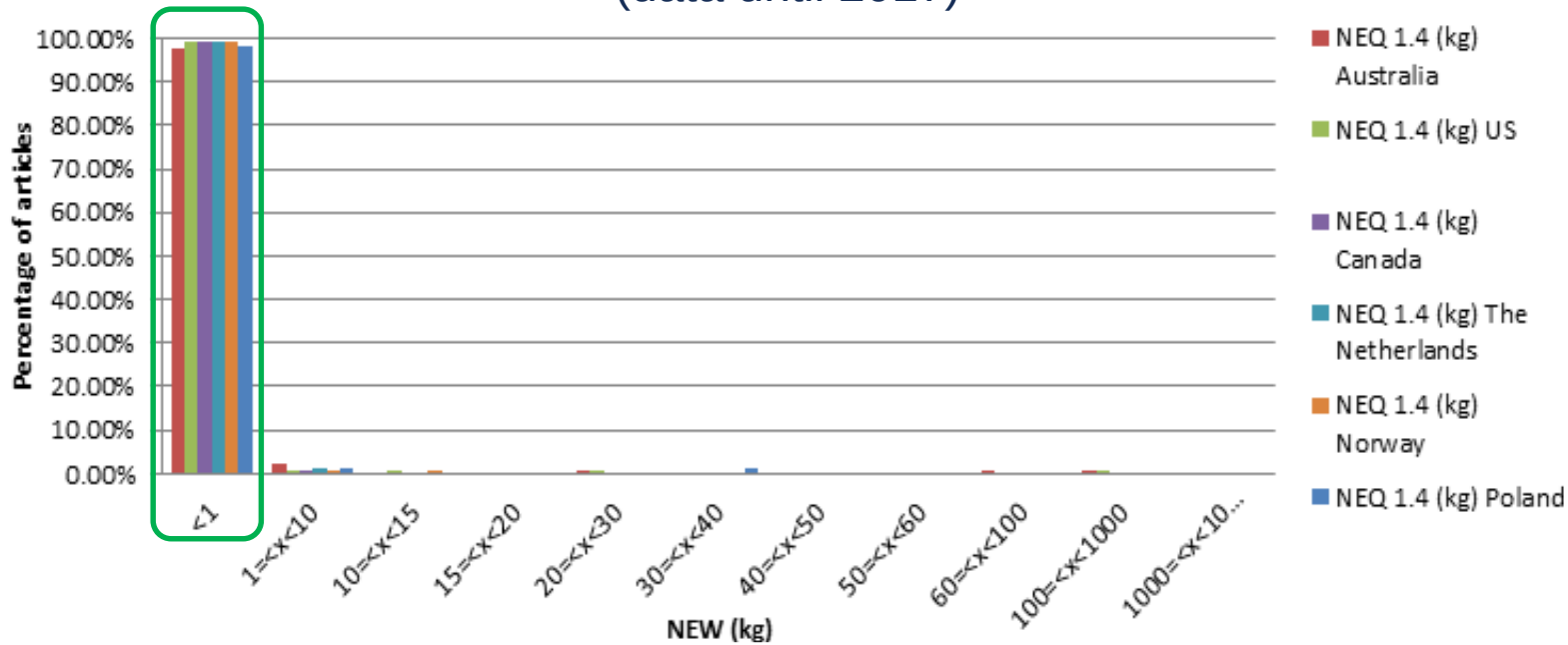
Percentage of HD assignment by nations
(data until 2017)



I. Hazard Classification in the Nations

- The analysis of the Net Explosive Quantity (NEQ) of HD 1.4 items shows that SAA and small items (fuzes, igniters) represent the vast majority of them.

NEQ for HD 1.4 assignments
(data until 2017)

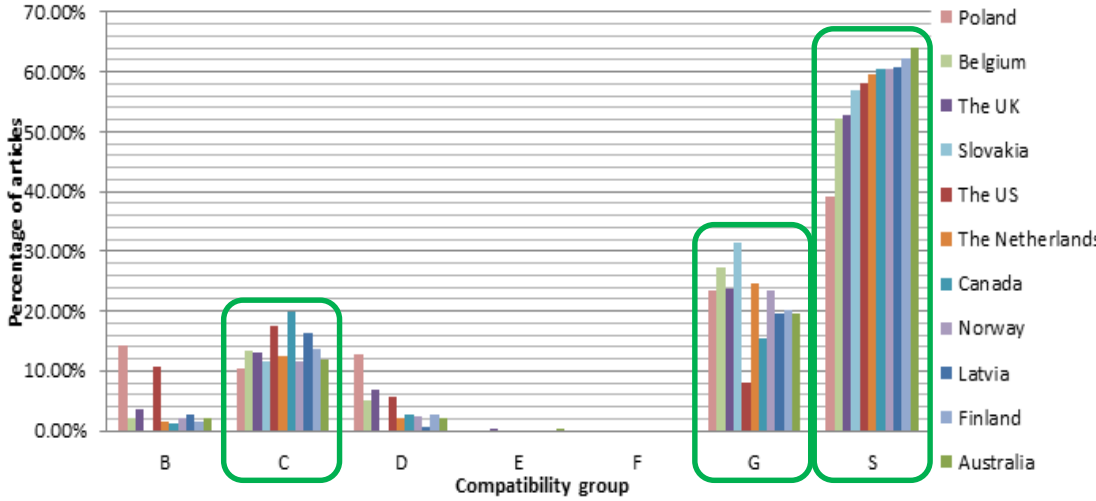


I. Hazard Classification in the Nations

- In almost all nations, more than 50% of HD 1.4 items are classified as HD 1.4 S.

- The other main CG used in HD 1.4 are:
  - CG C: Propellant explosive substance or other deflagrating explosive substance or article containing such explosive substance.
  - CG G: Pyrotechnic substance, or article containing a pyrotechnic substance, or article containing both an explosive substance and an illuminating, incendiary, tear- or smoke-producing substance (other than a water-activated article or one containing white phosphorous, phosphides, a pyrophoric substance, a flammable liquid or gel, or hypergolic liquids).

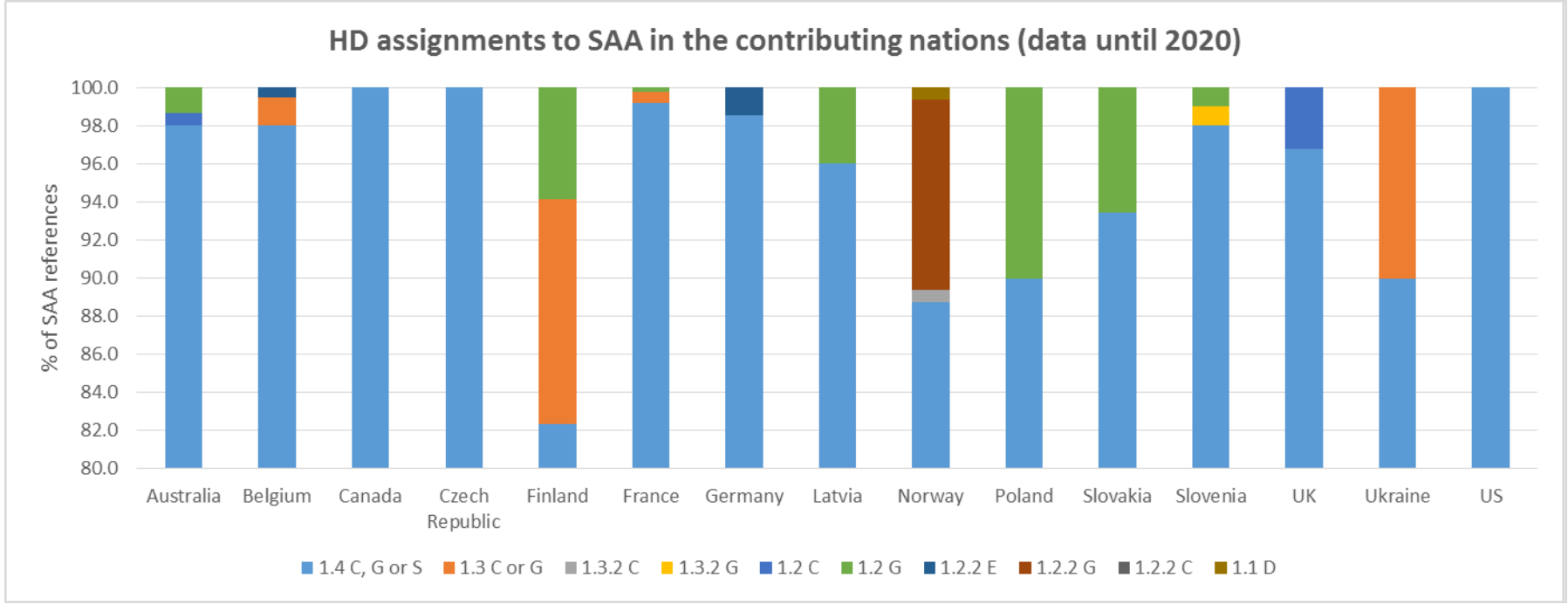
HC assignments by CG and nations for HD 1.4 (data until 2017)



II. Classification of SAA in the Nations

II. Classification of SAA in the Nations

- If we focus on SAA of .50 Cal. or less, the distribution in HD is as follows:



II. Classification of SAA in the Nations

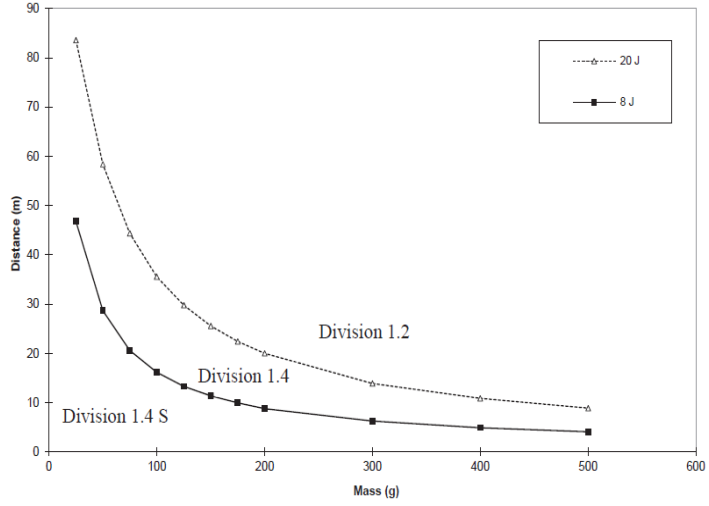
- The analysis of the previous chart shows that the great majority of SAA of calibre up to .50 in. (or 12.7 mm) are classified 1.4 C, G or S.

- The exceptions include:
  - Cartridges containing incendiary and/or tracer projectiles are sometimes classified **1.3(.2) C or G**
  - Cartridges containing incendiary and/or tracer projectiles are sometimes classified **1.2(.2) C, E or G**
  - One 12.7 mm reference is classified **1.1 D** (Norway)

- It can be noted that all SAA up to .50 are classified, without exception, 1.4 C, G or S in Canada, in Czech Republic, and in the US.

II. Classification of SAA in the Nations

- Some misclassifications and non-uniformities in HCC across nations were found during this analysis. A few examples:
  - The classification of a 12.7 mm MP in HD 1.1 seems unrealistic (as confirmed by Norway).
  - The compatibility groups C or G are usually assigned to munitions that do not pass the kinetic energy criterion to be included in 1.4 S → it seems unusual that SAA should be assigned to compatibility group D or E.



  - Some cartridges 12.7x99 mm classified in 1.2 G were purchased abroad and their HCC was accepted on face value.
  - 12.7 mm cartridges with bullet B-32 or BZT are classified **1.2 G** in Australia and in Poland, but they are classified **1.3 G** in Ukraine.

II. Classification of SAA in the Nations

- In the specific case of small arms ammunition (SAA), the US have a unique regulation.

- Two regulations for transportation of munitions co-exist:
  - TB 700-2 within the Department of Defence (DOD)
  - 49 CFR for a commercial venture with no DOD involvement

- Regarding SAA, these regulations indicate that cartridges and small arms may be assigned a classification code of 1.4S by the manufacturer provided they meet the following criteria:
  - Not a forbidden explosive
  - Ammunition for rifle, pistol, or shotgun
  - Ammunition with inert projectile or blank ammunition
  - Limitation in caliber:
    - **Not exceeding 50 caliber** for rifle or pistol cartridges or **8 gauge** for shotgun shells (in 49 CFR)
    - **Less than .50 caliber cartridges** including those used in machine guns or containing a small tracer in the projectile and shotgun shells **not exceeding 12 gauge** (in TB 700-2)

II. Classification of SAA in the Nations

- Moreover, the 2001 DOD memo states that:

  (…) the current inventory of Small Arms Ammunition and all future similar technology Small Arms Ammunition, with no incendiary or high explosive projectiles, ***shall be deemed IM-compliant****.*

- Also in Canada and France, SAA are often exempt of IM assessment. As stated in the French DGA instruction 1184:

  "*Munitions containing small quantities of active materials and not representing serious hazards in the logistic phase, i.e. potential classification in hazard division* ***HD 1.4*** *in accordance with the UN Manual of Tests and Criteria, are exempt from the* [IM] *process (…)*."

- These exemptions and default hazard classifications are convenient but they could lead to **under classification of SAA** if done without judgement (e.g. HC on face value, successive classifications by assimilation).
- Does it cover all situations?



II. Classification of SAA in the Nations

Key figures for a convoy transporting troops and munitions (with SAA being by far the most represented category, in number) in Mali



D. Perez, "Le Service interarmées des munitions (SIMu) French Joint Service for ammunition," in *3rd European IM Day*, Amsterdam, The Netherlands, May 2017

Conclusion

- The analysis of the National Hazard Classification database hosted by MSIAC for the nations showed that:
  - HD 1.4 articles are the most frequently occurring
  - For HD 1.4, CG S occurs the most, but also C and G.
- A more focused analysis on HCC for SAA showed that:
  - The great majority of SAA of caliber up to .50 in. (or 12.7 mm) are classified 1.4 C, G or S. 100% in USA, CAN and CZE.
  - SAA that are not classified in HD 1.4 are typically cartridges containing incendiary and/or tracer projectiles, which is justified.
  - Thanks to this analysis, it was possible to spot some misclassifications and non-uniformities in HCC across the nations.

- Classification by default and exemptions for SAA may lead to safety breaches. Why not revisit the classification of your SAA stockpile ?



U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Effects of Using Fluid Energy Milled HMX in LX-14

Presenter: John Latwinas

Team: Philip Samuels, David Soffer, Sean Swaszek, Erik Wrobel, David Rydzewski, Henry Grau, Alex Gandzelko, Caleb Owens, Dr. Daniel Iwaniuk, Joshua Felts (Indian Head)

CCDC DEVCOM; Picatinny Arsenal, NJ

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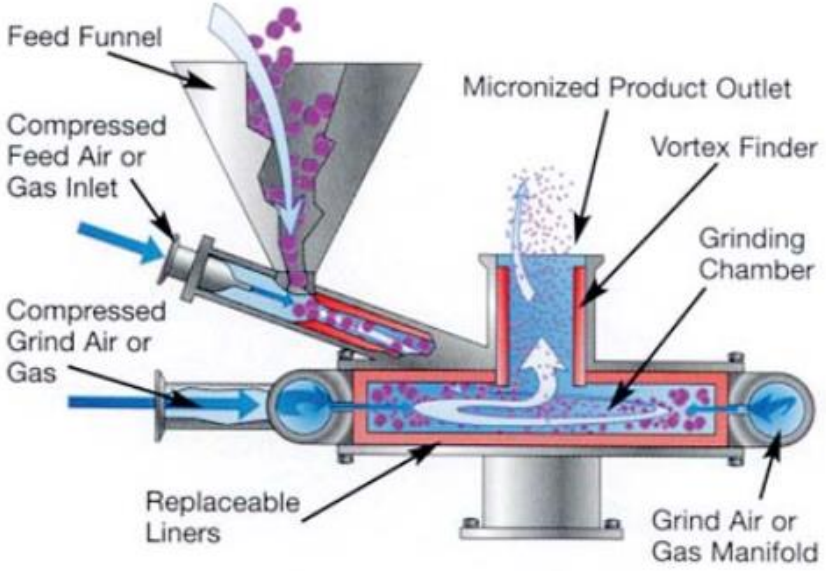
Distribution A

BACKGROUND

- LX-14 is a Lawrence Livermore National Laboratory developed explosive that is used in shape charge and main charge fill for the Hellfire missile, Javelin, TOW 2B, and other platforms.
- Due to its high nitramine content, it is considered a shock sensitive explosive and has witnessed a decline in use for more favorable IM explosives.
- Fluid energy milled (FEM) nitramines are currently being used in some qualified energetic formulations which have demonstrated reduced shock sensitivity in several investigations.
- FEM technology has led to the successful development of the IMX family of explosives, IMX-104 and PAX48.
- Coated Explosive Materials (CXM) also utilize FEM RDX for Navy and USAF products
- This study explores how safety testing and performance testing results of LX-14 made with FEM HMX compare to legacy LX-14 (made with larger granule HMX)

FLUID ENERGY MILL TECHNIQUE

- BAE Systems used a 4-inch Sturtevant Fluid Energy Mill (FEM) to grind HMX Class 1 to a reduced particle size.
- Fluid Energy Mill technology employs compressed air or gas to induce high-speed collisions between particles



- The final particle size range for FEM HMX has shown to 2-6 um[1].

[1] Brian Alexander. Characterization of LX-14 FEM and PBXN-9 FEM High Energy Explosives. BAE Systems. Holston Army Ammunition Plant Kingsport, Tennessee, USA. 2018 Insensitive Munitions & Energetic Materials Technology Symposium Portland, OR

TECHNICAL APPROACH

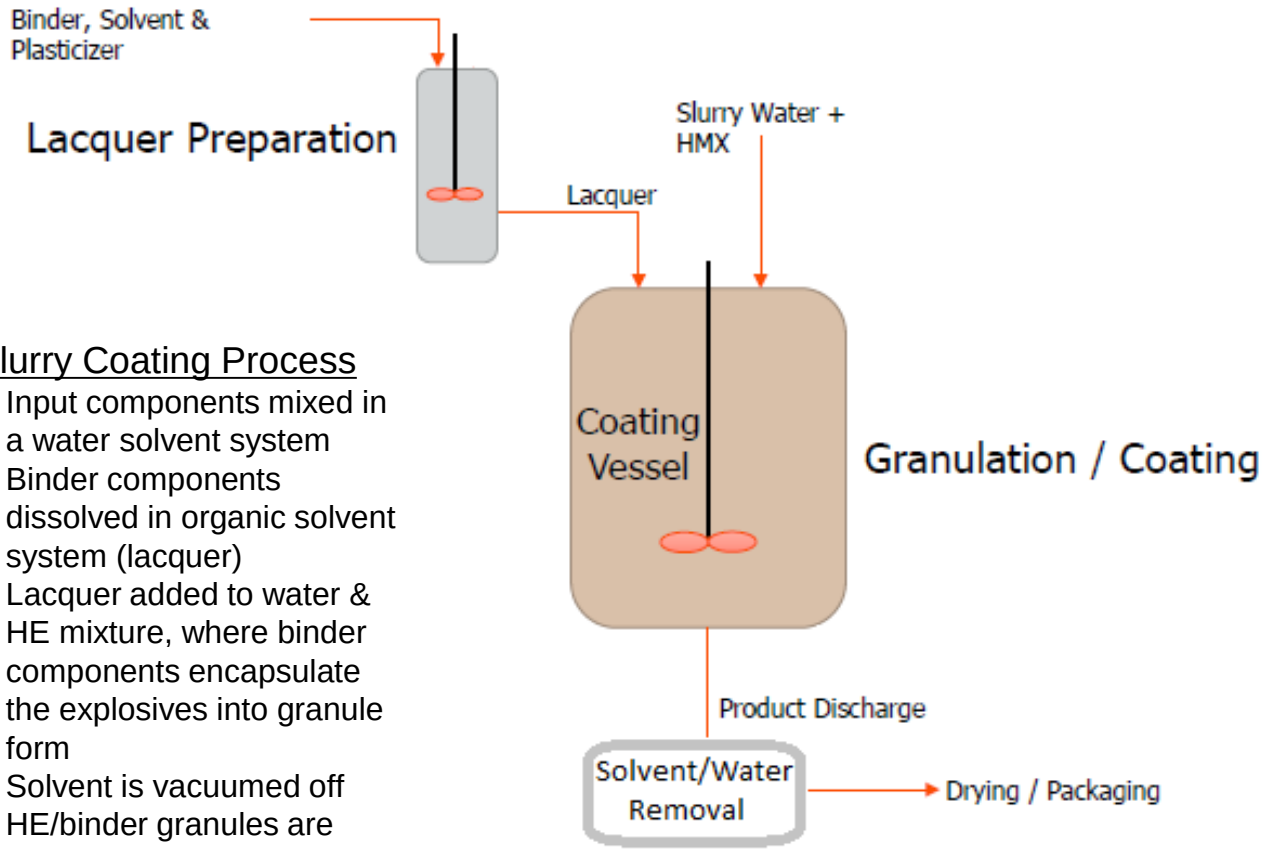
- LX-14 molding powder was prepared via slurry coating using the milled HMX





- The laboratory slurry coating process development was carried out in the Holston 10 Liter Coating Still.
- This laboratory still is similar in baffle arrangement and agitator design to the larger still used in manufacturing coating operations – so a smooth transition into production is to be expected

SLURRY COATING PROCESS



Slurry Coating Process

1. Input components mixed in a water solvent system
2. Binder components dissolved in organic solvent system (lacquer)
3. Lacquer added to water & HE mixture, where binder components encapsulate the explosives into granule form
4. Solvent is vacuumed off
5. HE/binder granules are filtered and dried

FORMULATIONS AND COMPOSITION ANALYSIS

- The legacy formulation of LX-14 is a proprietary mixture of HMX and Estane
- FEM HMX was utilized in LX-14 using traditional slurry coating process at 100% and 80%, the balance of which (20%) consisted of Class I HMX.
- The amount of FEM HMX was varied to find optimal shock sensitivity
- Certificates of Analysis from HSAAP were obtained for the FEM HMX batches, and the composition analysis results were deemed within the acceptable range of error

SAFETY TEST RESULTS







Formulation	ERL Impact (cm)	BAM Friction (N)	ABL ESD (J)
LX-14 (100% FEM HMX)	54.1	10/10 No Goes @ 360	20/20 No Goes @ 0.120
LX-14 (80% FEM HMX)	49.1	10/10 No Goes @ 324	20/20 No Goes @ 0.090
Legacy LX-14	24.9	10/10 No Goes @ 288	20/20 No Goes @ 0.020
FEM HMX	56.2	10/10 No Goes @ 144	20/20 No Goes @ 0.063
RDX Class I	26.1	10/10 No Goes @ 160	20/20 No Goes @ 0.020
HMX Class I	32.8	10/10 No Goes @ 108	20/20 No Goes @ 0.031

- ERL Impact results are H50 values

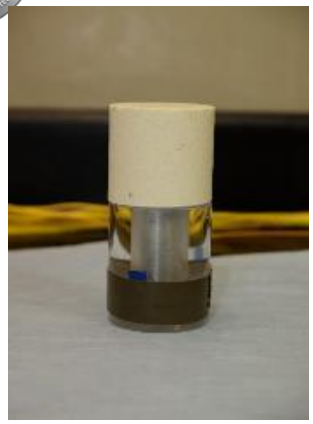
LARGE SCALE GAP TEST

Test	Special Conditions	Testing Facility	% FEM HMX	LSGT (Cards)	LSGT (kbar)	Density (g/cc)
1	None	Picatinny Arsenal	80	177-180	27.22-26.22	1.80-1.81
2	Aged 3 months at 70°C	Picatinny Arsenal	80	177-178	27.22-26.88	1.80-1.81
3	None	Picatinny Arsenal	80	166.5	31.1	1.81
4	None	Picatinny Arsenal	100	161.5	33.19	1.80
5	None	BAE Systems	100	176	27.56	1.67
6	Legacy LX-14	BAE Systems	0	236	14.47	1.784

- Above are the results for LSGTs of formulations with varied proportions of FEM HMX performed by BAE Systems and Picatinny Arsenal.
- MSIAC Newgates utilized for SGT card gap pressure conversion
- The significant density difference between the 100% FEM formulations was caused by the inability to heat the press tooling and pull vacuum at BAE during pellet pressing operations
- Results show significant improvement in insensitivity for formulations which incorporate FEM HMX, compared to legacy LX-14
- Pressing difficulties presented by the 100% FEM formulation indicate possible future production complications for that specific variant.



IHE GAP TEST





- The IHE Gap test was executed per US National Section of AOP-7 (Draft SRD-AOP-7.2) which uses a pressed pentolite explosive (density of 1.56 g/cm^3) donor system
- Test apparatus (above) consists of either two stacked pellets or one pellet (2.0 inches thick and 2.0 inches in diameter), each 1.0-inch thick and 2.0 inches in diameter
- Polymethylmethacrylate (PMMA) spacers are used as the attenuator material between the donor and acceptor charges.
- Utilized MSIAC Newgates for IHE Card Gap pressure conversion
- Results show an improvement in insensitivity when the HMX is milled for LX-14

Formulation	Density (g/cc)	Card Gap	Gap Pressure (kbar)
Legacy LX-14	1.82	225.5	15.93
LX-14 with 80% FEM HMX	1.80	169.5	29.93

LARGE SCALE DETONATION VELOCITY



- Large Scale Detonation Velocity (LSDV) Tests were performed on both formulations and compared to legacy LX-14 at Picatinny Arsenal
- The LSDV test setup is shown to the right.
- In a LSDV test, a booster detonates the main charge pressed into a LSGT tube. When the detonation front passes wires at the inlet and outlet, the times are recorded by a remote signal box on the other end of the wires. The detonation velocity is calculated using the time difference between the pulses and length of the LSGT tube

- Numerical results are shown in the table to the right.
- Both FEM formulations are comparable in performance to Legacy LX-14 and PBXN-9

Formulation	Density (g/cc)	Det. Vel. (mm/us)
LX-14 80 FEM	1.81	8.63
	1.81	8.65
LX-14 100 FEM	1.8	8.63
	1.8	8.63
Legacy LX-14	1.82	8.79
PBXN-9	1.74	8.4

SMALL SCALE FRAGMENT ATTACK RESULTS

- Small-Scale Fragment Attack (SSFA) tests were conducted to determine the reaction violence from a frag attack on LX-14 FEM HMX compared to legacy LX-14
  - ✓ **Result = LX-14 FEM HMX had an improved response compared to legacy LX-14**

Sample	Results w.r.t. Cover Plate Thickness (inches) for Single Liner in RP-4			
	1/4”	5/16”	3/8”	½”
LX-14 (80% FEM)	**Explosion** • No HE recovered	N/A	**Deflagration** • No HE recovered	**Deflagration** • No HE recovered
LX-14 (Legacy)	**Explosion** • No HE recovered	**Explosion** • No HE recovered	**Explosion** • No HE recovered	**Explosion** • 22.73g HE recovered



- Test setup (left)
- A deflagration (right) indicates that a reaction was violent enough to break the tube into a few pieces and break the bolts
- An explosion indicates cover plate perforated, tube split into pieces, rods and closure plate broken



TECHNICAL CONCLUSIONS

- Improved insensitivity and safety results of milled HMX formulations were observed

Formulation	ERL Impact (cm)	BAM Friction (N)	ABL ESD (J)	LSGT	IHE Gap
80% FEM LX-14	49.1	No rxn @ 324 N	No rxn @ 0.09 J	166.5 - 180 cards (31.1 - 26.22 kbar)	169.5 cards (29.93 kbar)
100% FEM LX-14	54.1	No rxn @ 360 N	No rxn @ 0.12 J	161.5 cards (33.19 kbar)	N/A
Legacy LX-14	24.9	No rxn @ 288 N	No rxn @ 0.02 J	236 cards (14.47 kbar)	225.5 cards (15.93 kbar)

- Both milled HMX formulations maintained comparable performance level of Legacy molding powders LX-14 and PBXN-9
  - 80% FEM: LSDV ~ 8.64
  - 100% FEM: LSDV = 8.63

QUESTIONS?

Participants

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Dr. Daniel Iwaniuk
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U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Evaluation of PBXN-9 Utilizing FEM HMX

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(BAE Systems) Mr. Brian Alexander - R&D Sr. Principal Chemist

DEVCOM AC; Picatinny Arsenal, NJ
BAE SYSTEMS

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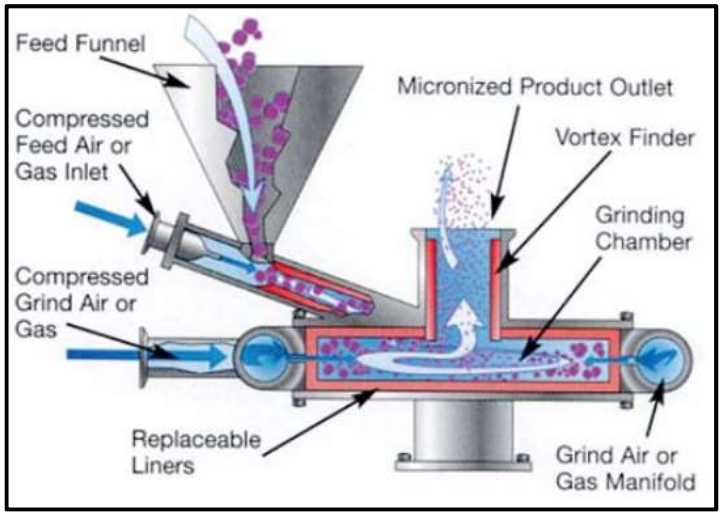
07 – 08 APRIL 2021

BRIEFING OUTLINE

1. Background & Technical Description
2. PBXN-9 FEM HMX Characterization & Pressing
3. Testing and Evaluation
    a) Performance Testing
    b) Shock Sensitivity Testing
    c) Mechanical Properties Testing
    d) Small-Scale Fragment Attack Testing
    e) Setback Survivability Testing
    f) Irreversible Growth Study
    g) Exudation Study
4. Conclusions

BACKGROUND & TECHNICAL DESCRIPTION

- PBXN-9 Formulation:
  - HMX
  - DOA (dioctyl adipate) as plasticizer
  - HyTemp as binder
- Fluid Energy Mill (FEM) Technology:
  - Utilizes compressed air to grind particles to less than 10 microns in size
- FEM nitramines are currently used in qualified energetic formulations:
  - Properties have not yet been fully characterized
  - FEM RDX is currently in full rate production, however, FEM HMX has not been fully explored



- BAE Systems has produced FEM HMX formulations:
  - LX-14 with 80% and 100% FEM HMX
  - PBXN-9 with 100% FEM HMX
  - Formulations have been successfully scaled-up for LSGT and performance testing

https://bulkhand.com/2014/08/sturtevant-proven-size-reduction-classifying-technology/
http://www.jbmprispac.com/pd/15/air-jet-mill

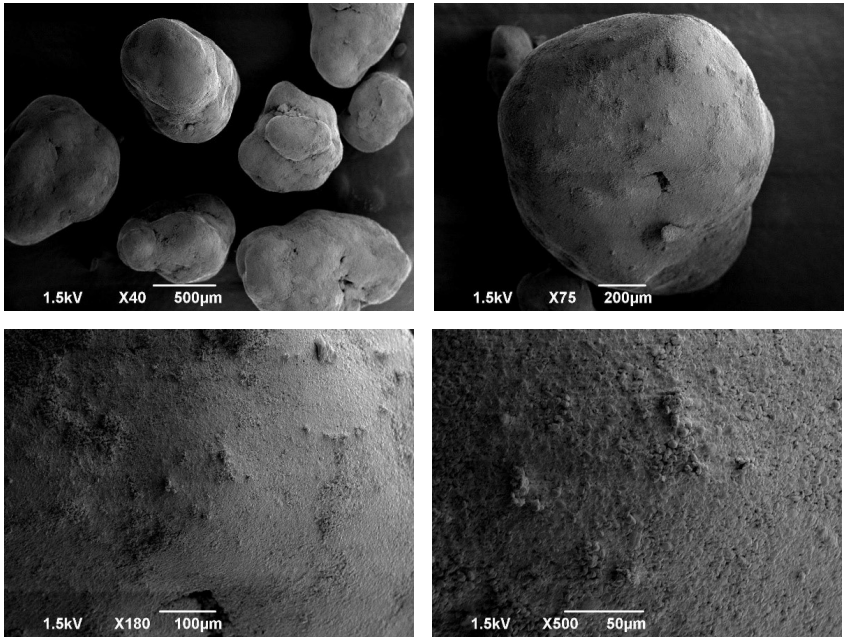
PBXN-9 FEM HMX: CHARACTERIZATION

➢ Initial Safety Testing:

Material	ERL Impact (cm)	BAM Friction (N)	ESD (J)
PBXN-9 FEM HMX	27.7	10/10 No Goes @ 216	20/20 No Goes @ 0.020
Legacy PBXN-9	40.9	10/10 No Goes @ 324	20/20 No Goes @ 0.020
RDX (Class I Type II)	27.2	10/10 No Goes @ 168	20/20 No Goes @ 0.040

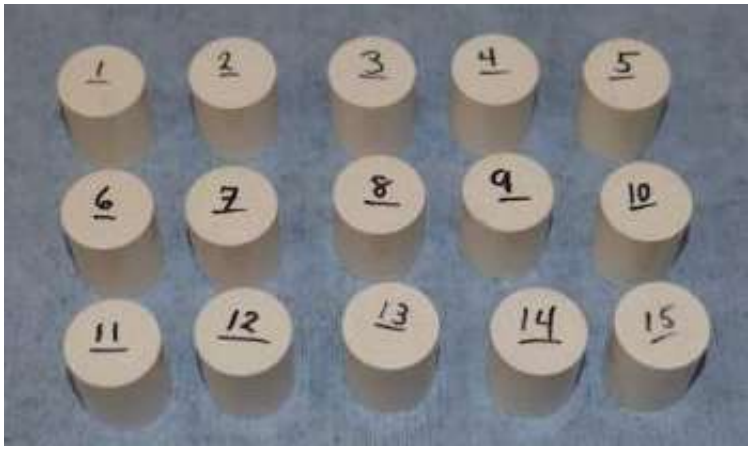
➢ SEM Images of Molding Powder:

- ✓ Granules were well coated and spherical in shape



➢ Pressing Study:

- ✓ Consistently achieved 98.5% theoretical maximum density (TMD)



PBXN-9 FEM HMX: LARGE SCALE DETONATION VELOCITY TEST

- A Large Scale Detonation Velocity (LSDV) test was conduct to determine the detonation velocity of PBXN-9 FEM HMX compared to legacy PBXN-9
  - ✓ **Result = PBXN-9 FEM HMX detonation velocity is equivalent to legacy PBXN-9**

Formulation	Density (g/cc)	DV (km/s)	Dent (inches)
PBXN-9 with 100% FEM HMX	1.72	8.41	0.382
PBXN-9 with 100% FEM HMX	1.72	8.40	0.369
Legacy PBXN-9	1.64	8.07	0.378
Legacy PBXN-9	1.64	8.11	0.376
Legacy PBXN-9	1.70	8.33	0.400
Legacy PBXN-9	1.70	8.30	0.403
Legacy PBXN-9	1.74	8.46	0.417
Legacy PBXN-9	1.74	8.48	0.426



PBXN-9 FEM HMX: LARGE SCALE GAP TEST

- A Large Scale Gap Test (LSGT) was conduct to determine the shock sensitivity of PBXN-9 FEM HMX compared to legacy PBXN-9
  - ✓ **Result = PBXN-9 FEM HMX showed a 37 – 54 card improvement compared to legacy PBXN-9**

Formulation	Testing Facility	Percent of FEM HMX	LSGT (Cards)	LSGT (Kbar)	Density (g/cc)
PBXN-9 FEM HMX	Picatinny Arsenal	100	167.5	30.70	1.72
Legacy PBXN-9	Picatinny Arsenal	0	222.0	16.47	1.70
Legacy PBXN-9	Picatinny Arsenal	0	205.0	19.54	1.74
Legacy PBXN-9	BAE Systems	0	186.5	24.22	1.69
PBXN-9 FEM HMX	BAE Systems	45	184.5	24.81	1.67
PBXN-9 FEM HMX	BAE Systems	75	186.0	24.36	1.65
PBXN-9 FEM HMX	BAE Systems	100	156.0	35.69	1.65



PBXN-9 FEM HMX: UNI-AXIAL COMPRESSION TESTING

- A series of uni-axial compression tests were conduct at different temperatures to determine if utilizing 100% FEM HMX improved the mechanical strength of the PBXN-9 formulation
  - ✓ **Result = PBXN-9 FEM HMX showed an increase in strength, modulus, and strain at peak pressure compared to legacy PBXN-9**

PBXN-9 FEM HMX: SMALL-SCALE FRAGMENT ATTACK TEST

- Small-Scale Fragment Attack (SSFA) tests were conducted to determine the reaction violence from a frag attack on PBXN-9 FEM HMX compared to legacy PBXN-9
  - ✓ **Result = PBXN-9 FEM HMX had an improved response compared to legacy PBXN-9**

Sample	Results w.r.t. Cover Plate Thickness (inches) for Single Liner in RP-4			
	1/8" (3.2 mm)	1/4" (6.3 mm)	3/8" (9.5 mm)	1/2" (12.7 mm)
PBXN-9 FEM HMX	Detonation	No Reaction Nearly all HE remained No Reaction Nearly all HE remained	No Test	No Test
PBXN-9 (Legacy)	Detonation	Deflagration	No Reaction	No Test

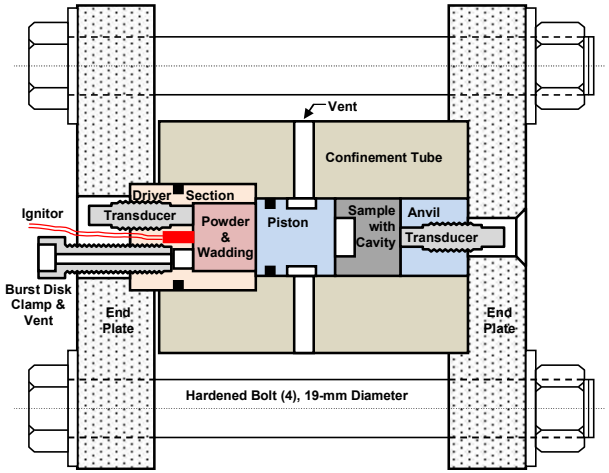


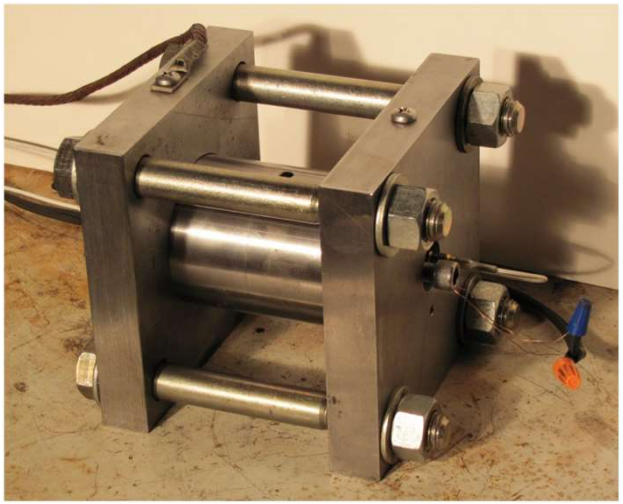
Levels of Reaction	
No Reaction	Apparatus intact with <1% tube expansion
Burn	Apparatus intact with tube bulged or having a single split; alternatively, rods broken with <1% tube expansion
Deflagration	End closures undamaged except for possible broken edges on Closure Plate; Tube split into pieces or split and opened wide, either which can break rods; Alternatively, tube just bulged and Cover Plate or rods broken to release pressure
Explosion	Clamp & Spacer broken, Cover Plate perforated but not sheared through opening in Clamp & Spacer, Tube split into pieces, Rods broken, Closure Plate broken with center intact
Detonation	Clamp & Spacer broken into pieces, Cover Plate sheared through opening in Clamp & Spacer, Tube and Closure Plate fragmented, no sample recovered

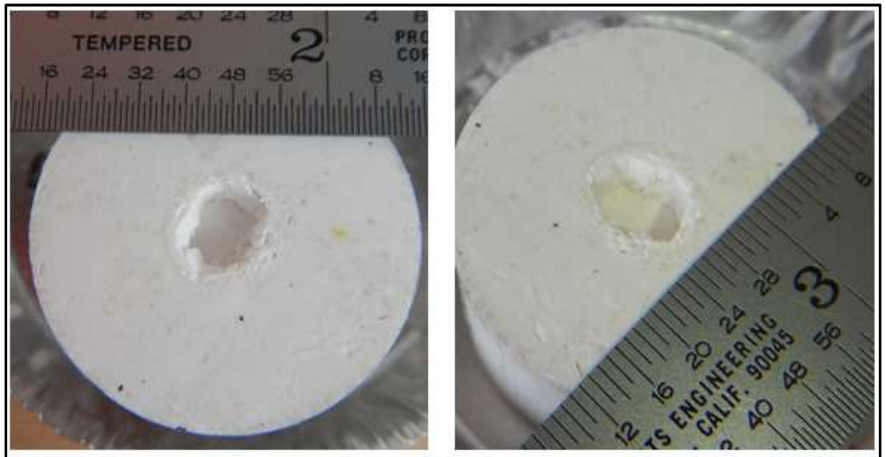


PBXN-9 FEM HMX: NSWC SETBACK RESULTS

- NSWC Setback testing was conducted on PBXN-9 FEM HMX as a screening tool for setback sensitivity
  - ✓ **Result = PBXN-9 FEM HMX showed improved response compared to legacy PBXN-9**





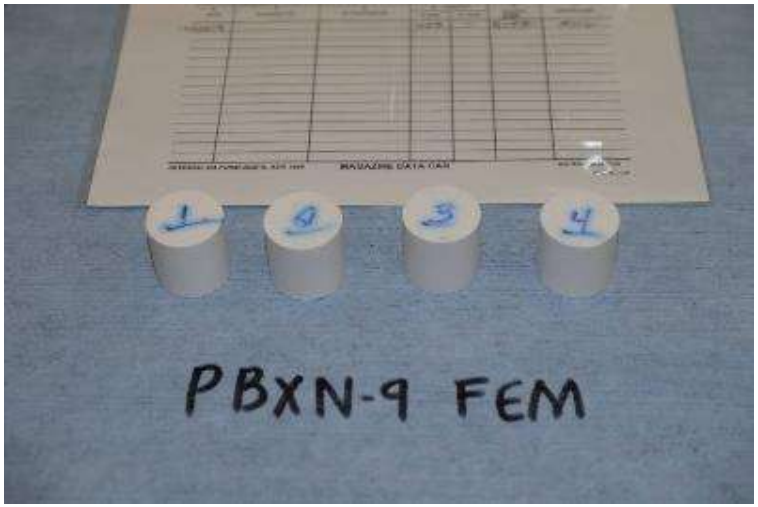


Sample Pellet - Post-Test
(No Reaction, Partial Cavity Collapse)

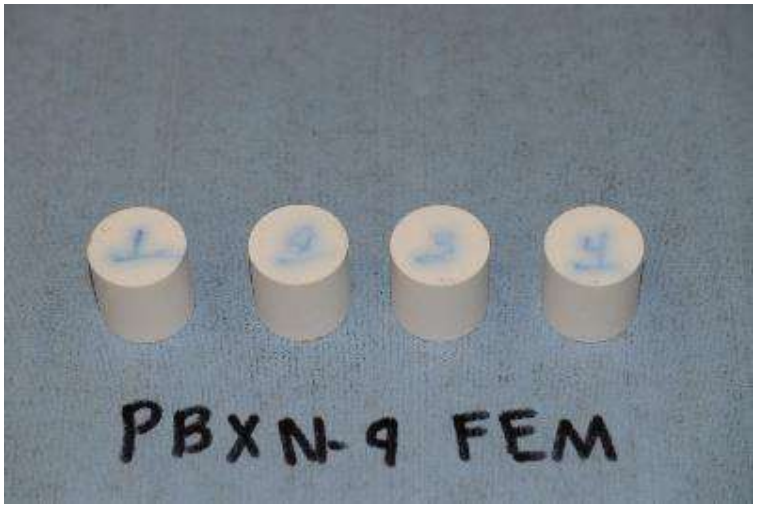
PBXN-9 FEM HMX: IRREVERSIBLE GROWTH STUDY

- Irreversible Growth testing was conducted to determine if PBXN-9 FEM HMX pellets increased in volume after thermal cycling
  - ✓ **Result = PBXN-9 FEM HEM showed no notable volume changes after thermal cycling; PASSED with no more than 1% volume change**

PBXN-9 FEM	Δ Volume (%)
1	-0.9565
2	-1.0258
3	-1.1548
4	-0.7295
Average	**-0.9666**



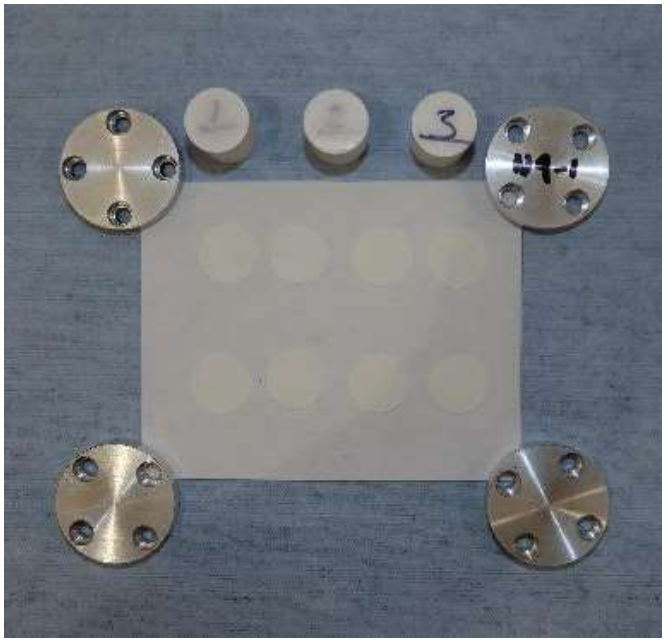
30 Temp Cycles
-54°C to 60°C
Dwell 4 hrs @ Extremes



PBXN-9 FEM HMX: EXUDATION STUDY

- Exudation testing was conducted to determine if the binder system exudates from pressed PBXN-9 FEM HMX after being exposed to elevated temperatures
  - ✓ **Result = PBXN-9 FEM HEM showed no evidence of exudation**

Test Cylinder	Sample Mass Loss (g)	% Exudation (Sample)	Filter Paper Mass Gain (g)	% Exudation (Filter Paper)
1	0.0411	0.0376	0.0161	0.0147
2	0.0238	0.0218	0.0169	0.0155
3	0.0357	0.0326	0.0240	0.0219



CONCLUSIONS

- ✓ A PBXN-9 analog utilizing Fluid Energy Milled (FEM) HMX was successfully developed, characterized, and evaluated as a collaborative effort between BAE Systems and DEVCOM AC

- ✓ The PBXN-9 FEM HMX formulation had equivalent performance compared to legacy PBXN-9
  - PBXN-9 FEM HMX: DV = 8.41 km/s (d = 1.72 g/cc)
  - Legacy PBXN-9: DV = 8.47 km/s (d = 1.74 g/cc)

- ✓ The PBXN-9 FEM HMX formulation had improved shock sensitivity, setback survivability, and mechanical properties compared to legacy PBXN-9:
  - LSGT testing resulted in 167.5 cards (legacy PBXN-9 LSGT = 205.0 to 222.0 cards)
  - Improved response in the SSFA test
  - Showed an improved response in the NSWC Setback Simulator test
  - Increase in strength, modulus, and strain at peak pressure

ACKNOWLEDGEMENTS & TEAM MEMBERS

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BAE Systems
Brian Alexander

Indian Head
Joshua Felts
Philipp Baldovi

QUESTIONS?

U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

Modeling of the M795 155 mm Artillery Projectile Sympathetic Reaction Test

Presentation to the 2021 Insensitive Munitions & Energetic Materials (IMEM) Technology Symposium

4/7/2021 – 4/8/2021

Erik Tolmachoff, PhD and Ryan Conner

Senior Engineers

CS Squared LLC

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INTRODUCTION

- **Problem: Model M795 155 mm artillery projectile sympathetic reaction test in 3D**
  - Donor and acceptor filled with either TNT or IMX-101 explosive fills
  - Used Ignition and Growth (I&G) reactive flow model for the acceptor explosive fill
  - Calculations performed in both CTH (SNL, finite difference) and ALE3D (LLNL, finite element)
  - Performed on workstation with 56 core (total) Intel processors with 2TB of RAM

- **Challenge: Obtaining a solution in a reasonable amount of time**
  - Literature for reactive flow material models typically recommend mesh resolutions >50 elements/cm
  - For full-scale 3D problems, such a high mesh resolution is computationally expensive in terms of memory and reduced time step for explicit codes (Courant limit)
  - Typical full-scale munition models would likely require scarce supercomputer resources

- **Approach: Reduce mesh resolution to allow solution in a reasonable timeframe**
  - Allows for complete runs in a few weeks of wall-clock time on an engineering workstation computer
  - Verify reduced level of mesh resolution chosen does not result in unsatisfactory error

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IGNITION AND GROWTH (I&G) REACTIVE FLOW MODEL

- **Developed in the 1970s and 80s by Lee and Tarver, includes three terms describing:**
  - Hot spot formation due to compression
  - Growth of hot spots due to formation of gas products
  - Slow final production of solid products

- **Able to capture complex reaction behavior from ignition through detonation including:**
  - 2D and 3D initiation and failure
  - Detailed propagation (corner turning)
  - Reactions from complex stimuli, including: 1) short durations shocks; 2) reflected shocks; 3) multiple shocks; and 4) ramp waves

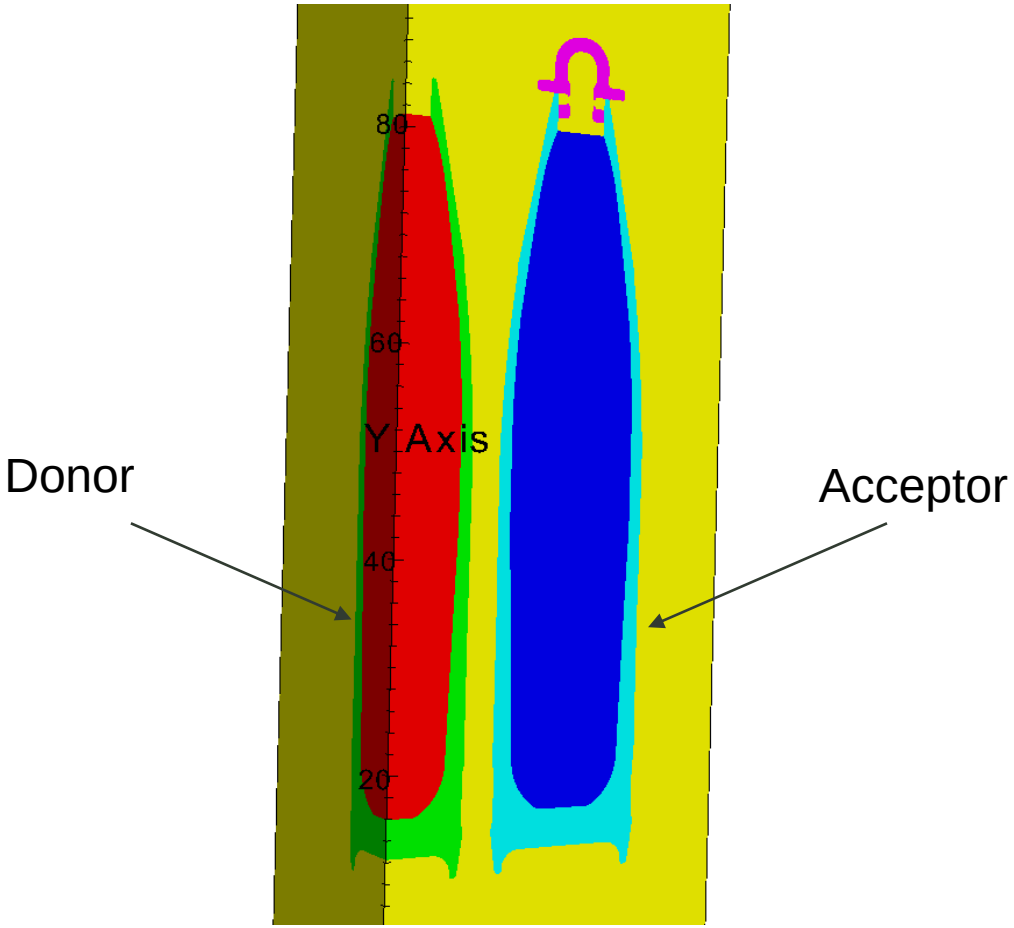
- **Simpler models cannot capture these complex responses → I&G is relatively simple and inexpensive reactive flow model**
  - JWL equations of state for legacy, new explosives are continuously developed and updated
  - There is a large library of I&G models

- **Potential difficulty is literature recommendation for highly refined mesh**
  - Important for excellent relative agreement when developing/validating models
  - Typically varies from 50 to several 100 elements/cm, depends on material (reaction zone thickness)
  - However, solutions may asymptote to mesh-independence at lower-than-recommended resolutions

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PROBLEM GEOMETRY, DOMAIN





Two unconfined projectiles
Computational domain:
(50 x 100 x 25 cm), ¼ symmetry along YZ plane, ½ symmetry along XY plane, outflow on all other boundaries
The “donor” is detonated, the “acceptor” experience a shock wave from the donor

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PROBLEM SETUP: ALE3D

- **Donor and acceptor were *both* filled with *either* TNT *or* IMX-101.**

- **Mesh is made by**
  - First hex meshing the entire computational domain.
    - Portion of acceptor that initially experiences shock is refined up to 25 elements/cm.
    - Ratioing is used to coarsen mesh away from the initial shock region of the acceptor.
  - Donor and acceptor parts are then shaped in.

- **I&G models for TNT and IMX-101 provided by LLNL.**
- **Projectile shells modeled using 4340 steel EOS and strength models from ALE3D library.**

- **Donor undergoes constant detonation velocity using programmed burn with JWL EOS at t=0.**

- **Problem run for up to 120 μs.**

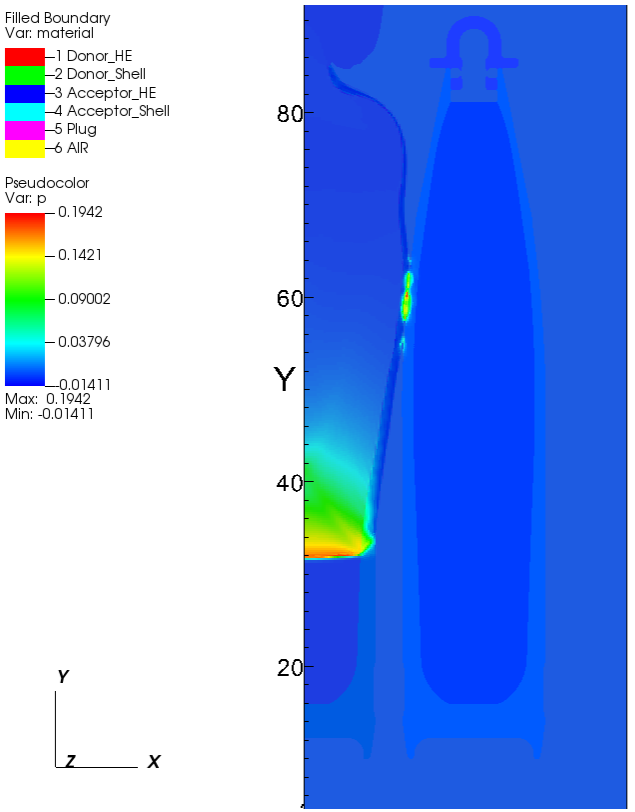
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PROBLEM SETUP: CTH

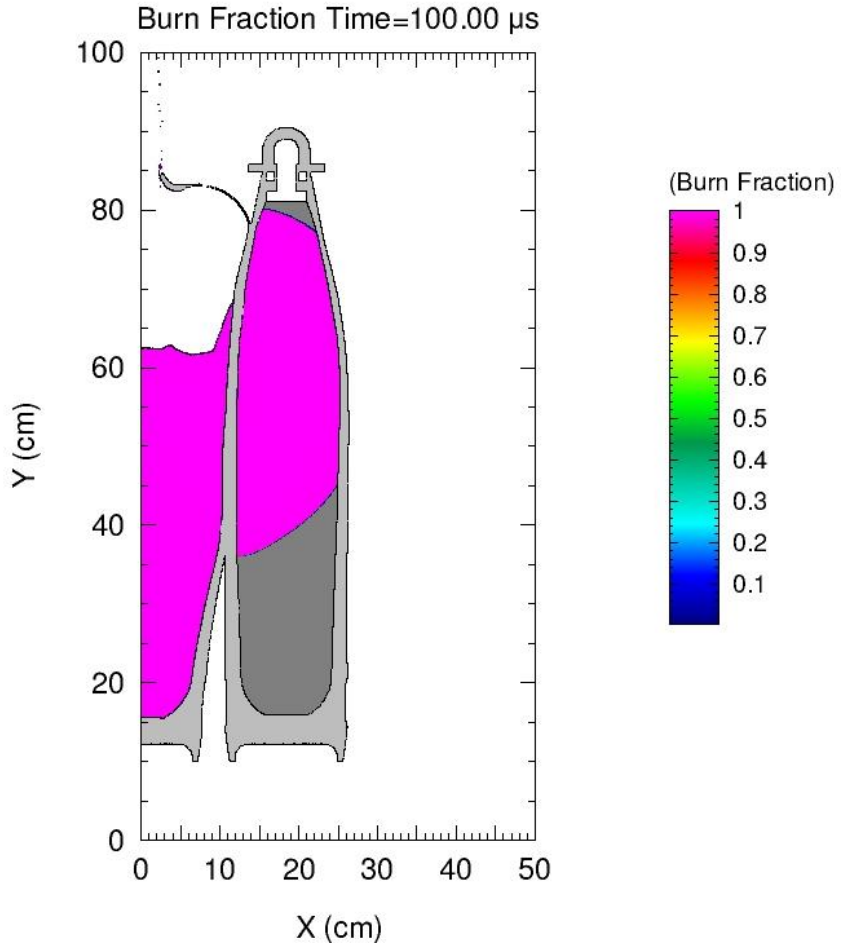
- **Donor and acceptor were *both* filled with *either* TNT *or* IMX-101.**
- **Model utilizes CTH's adaptive mesh refinement (AMR) algorithm to allow a mesh resolution of up to 25 elements/cm near detonation/shock waves and material interfaces.**
- **I&G models for TNT and IMX-101 provided by LLNL.**
- **Projectile shells modeled using 304 stainless steel EOS and strength models from CTH (12.2) library.**
- **Donor initiated with a programmed burn in small portion (similar to a booster), then transitions to a reactive flow model for the remainder of the donor reaction.**
- **Problem run for up to 100 μs.**

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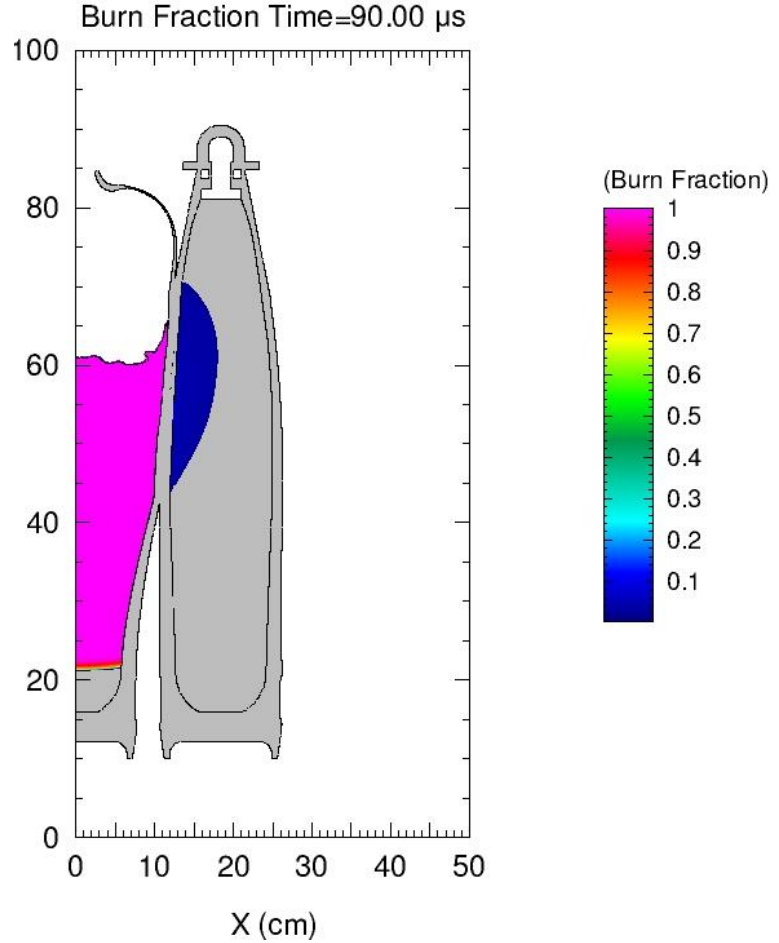
SIMULATION SNAPSHOTS OF 3D M795 PROJECTILE SYMPATHETIC REACTION



ALE3D solution at 72 μs, shortly after donor shell impacts acceptor. Plot of pressure contours (Mbar).



CTH solution for TNT filled M795s. TNT is a sensitive and the acceptor completely reacts.



CTH solution for IMX-101 fill. IMX-101 is less sensitive and the acceptor does not undergo extensive reaction.

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3D SYMPATHETIC REACTION RESULTS

- **Importantly, all the simulations run showed agreement with the tests. TNT-filled M795 projectiles sympathetically detonate while IMX-101-filled M795 projectiles do not.**

Table 1. ALE3D M795 sympathetic reaction runtime summary.

HE	Max Mesh resolution (#/cm)	Elements (millions)	Generation wall-clock time (hrs)	Run wall-clock time (hrs)
TNT	12.5	14	58*	16
TNT	12.5	3.89	22*	4
TNT	5	0.92	3*	1
TNT	25	52	302*	114
IMX-101	25	52	243	132

Table 2. CTH M795 sympathetic reaction runtime summary.

HE	**Max Mesh Resolution (#/cm)**	**Simulation End Time (μsec)**	**Wall-Clock Time (hrs)**
TNT	25	100	379
IMX-101	25	90	242

- **Bottom line: we can successfully simulate sympathetic reactions that agree with tests with reasonable turnaround of ~2 weeks on an engineering workstation.**

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2D EXAMINATION OF MESH RESOLUTION

- **Reduced mesh resolution 3D models presented provide results that agree with tests, now we want to assess how the mesh resolution affects solutions using these specific I&G material models.**
- **Approach: Model 1D and 2D flyer plate impact of TNT and IMX-101 using I&G and vary mesh resolution in both ALE3D and CTH**
- **Flyer plate impact experiments are commonly used to develop/validate reactive flow models including I&G.**
- **The goal is to perform simulations with impact velocity and pressure stimuli comparable to those experienced during sympathetic reaction and flyer plate testing**



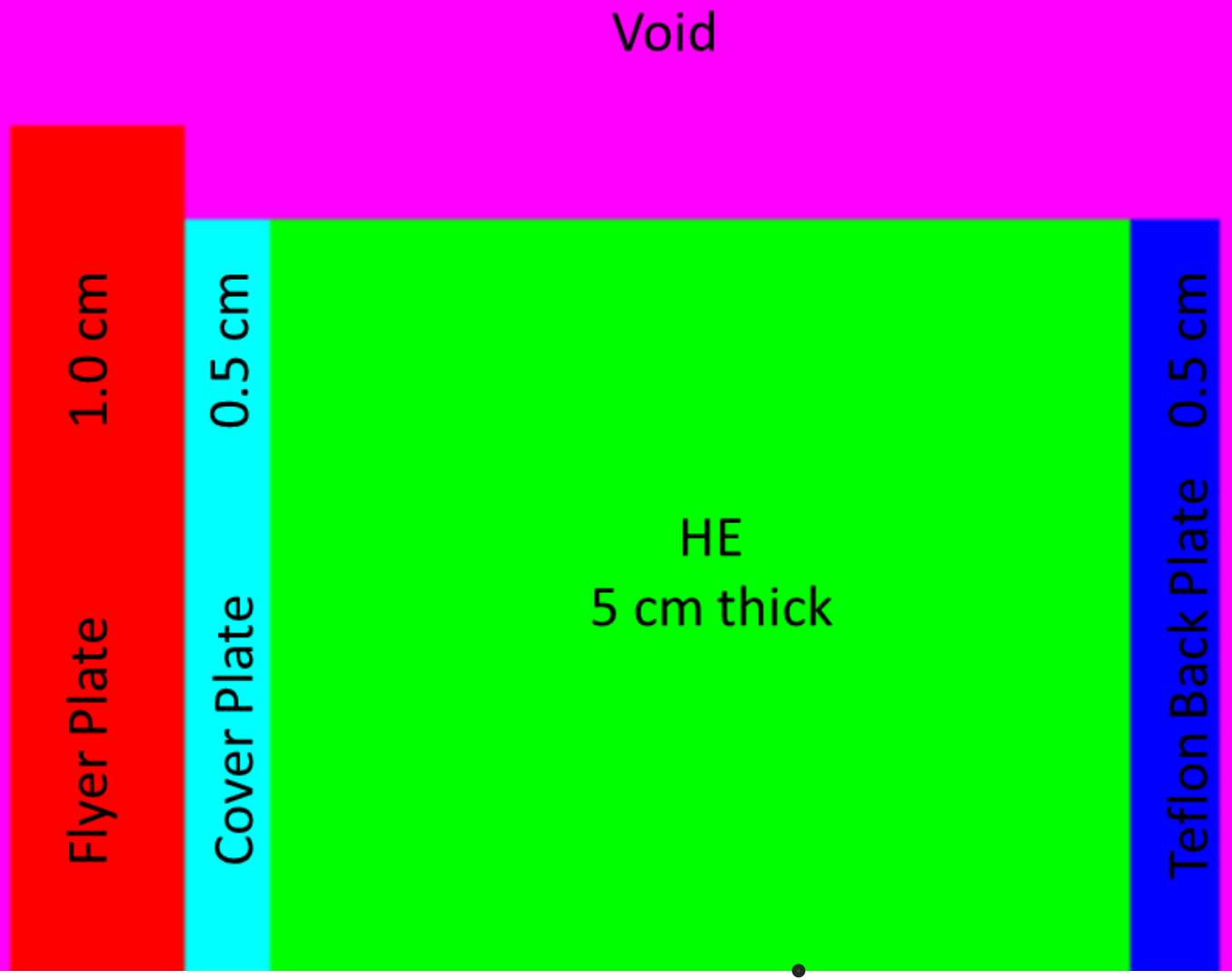
Example of a flyer plate experimental target. A sandwich consisting of a cover plate and a several layers of explosive are instrumented with pressure gauges. A plate with a diameter greater than the target strikes the experiment at a high velocity. Recorded data can be used to develop and validate reactive flow models.

Source: On the low pressure shock initiation of octahydro-1,3,5,7–tetranitro-1,3,5,7-tetrazocine based plastic bonded explosives, Vandersall, et al., J. App. Phys, 107, 094906 (2010)

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2D FLYER PLATE SETUP: ALE3D

- Flyer plate, cover plate are 4340 steel
- HE is TNT or IMX-101
- Axisymmetric @ y=0 boundary
- Outflow on all other boundaries
- Flyer plate is prescribed an initial velocity [800-1800m/s]
- Pressure and reacted fraction are traced in the HE.
- Three nominal mesh resolutions: 12.5, 25, 50 elements/cm

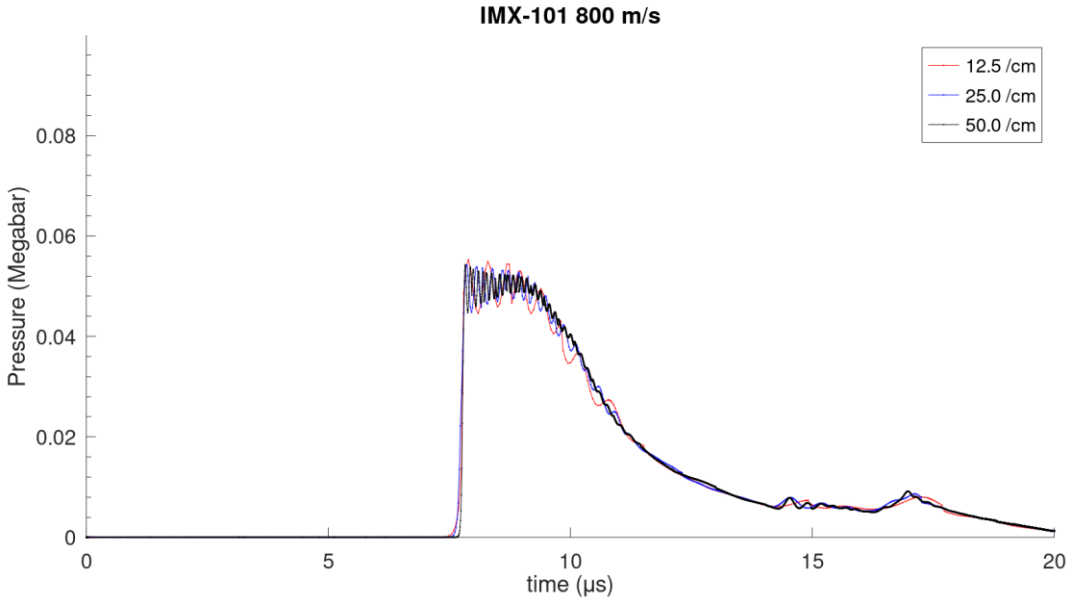


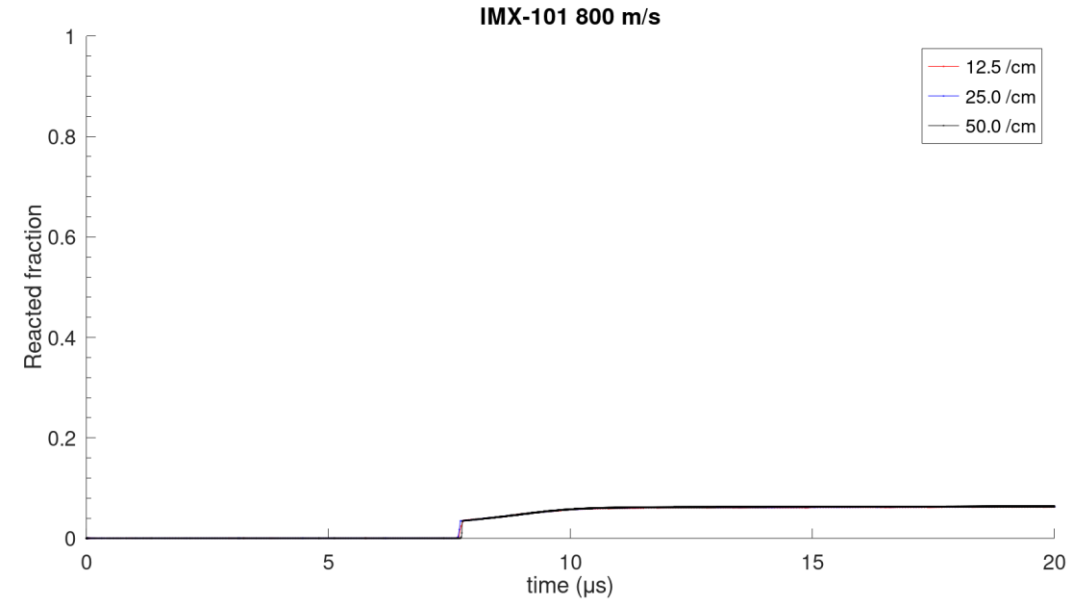
Virtual tracer 3 cm from the records pressure, reacted fraction

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ALE3D 2D RESULTS IMX-101: 800 M/S IMPACT VELOCITY





ALE3D results for IMX-101 with a flyer plate velocity of 800 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

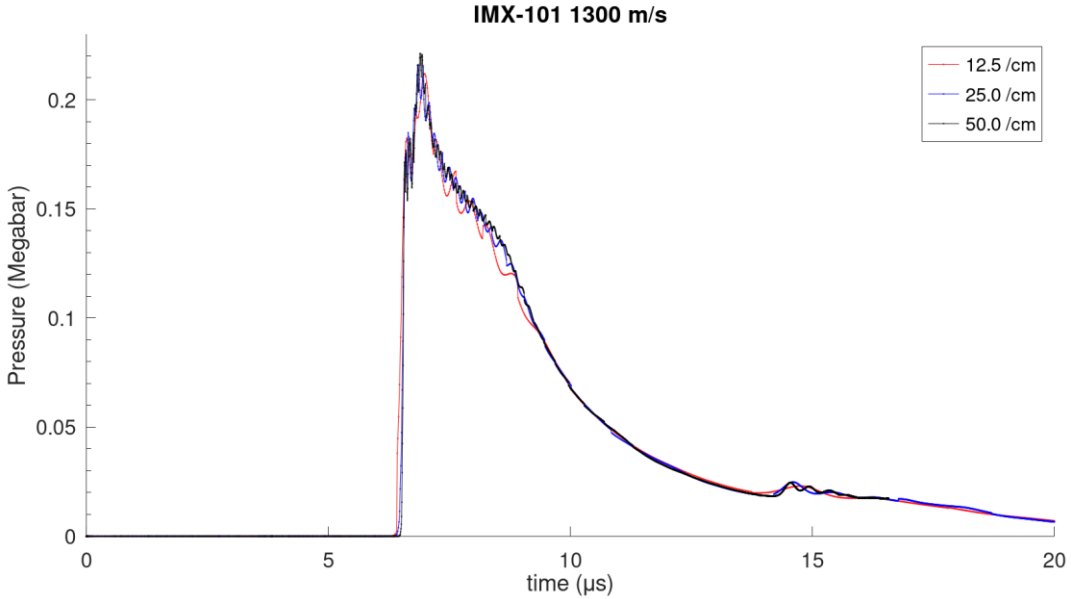
The tracer indicates that IMX-101 only reacts to limited extent.

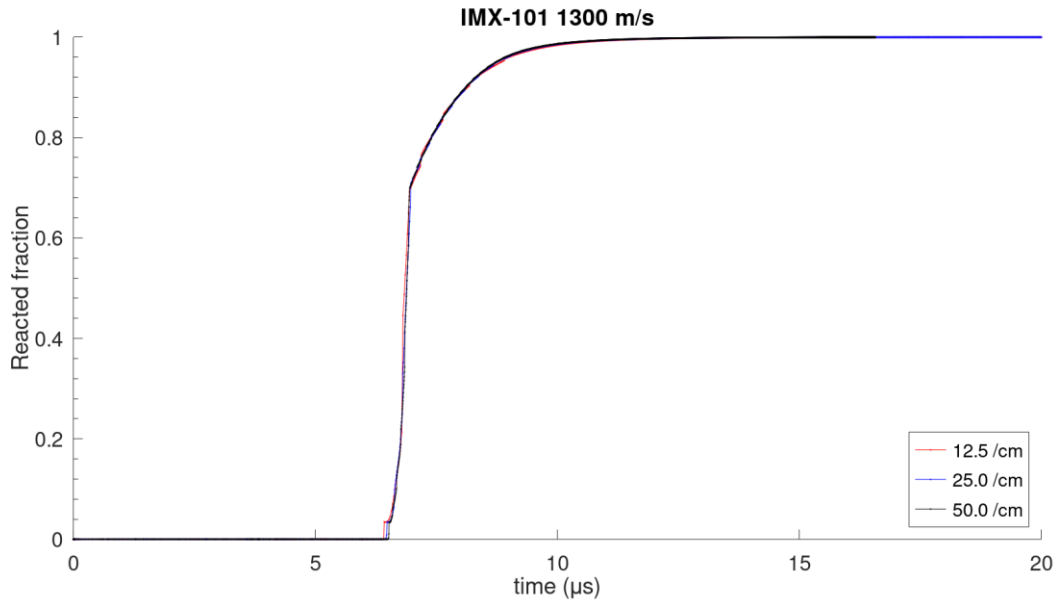
A close examination of the results shows that the solution asymptotes towards a mesh-independent solution at a resolution ~25 elements/cm

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ALE3D 2D RESULTS IMX-101: 1300 M/S IMPACT VELOCITY





ALE3D results for IMX-101 with a flyer plate velocity of 1300 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

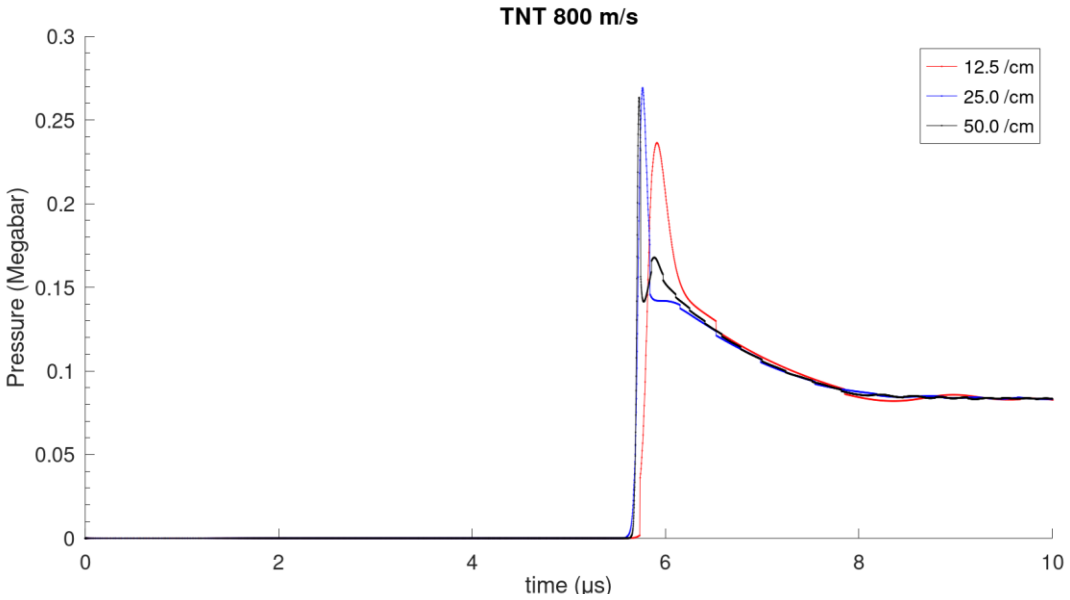
The tracer indicates that IMX-101 is detonating at the time the reaction passes through it.

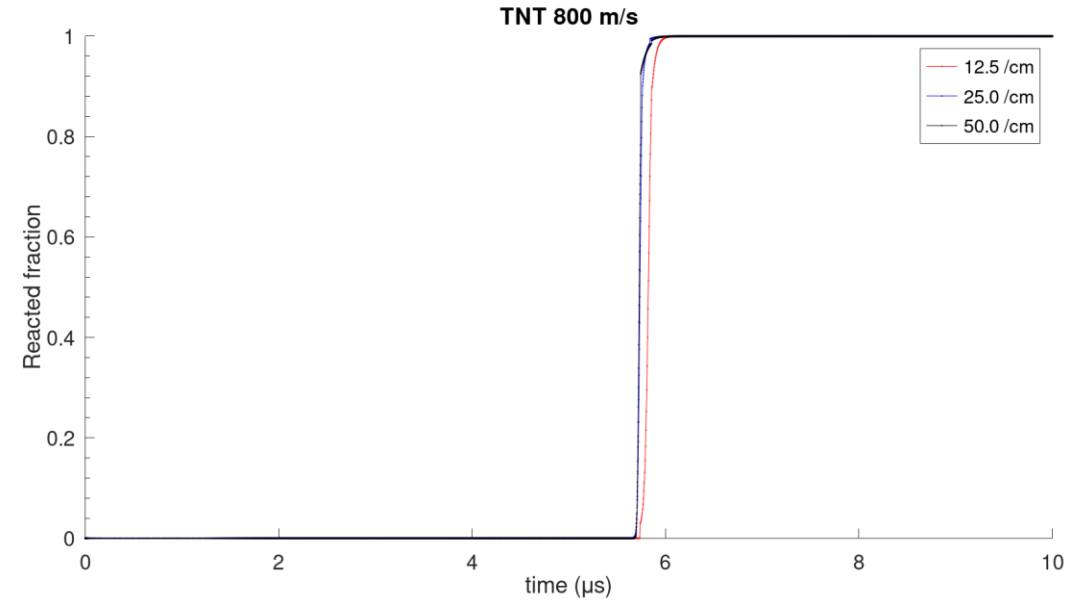
The results show very good agreement across the three levels of mesh resolution.

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ALE3D 2D RESULTS TNT: 800 M/S IMPACT VELOCITY





ALE3D results for TNT with a flyer plate velocity of 800 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

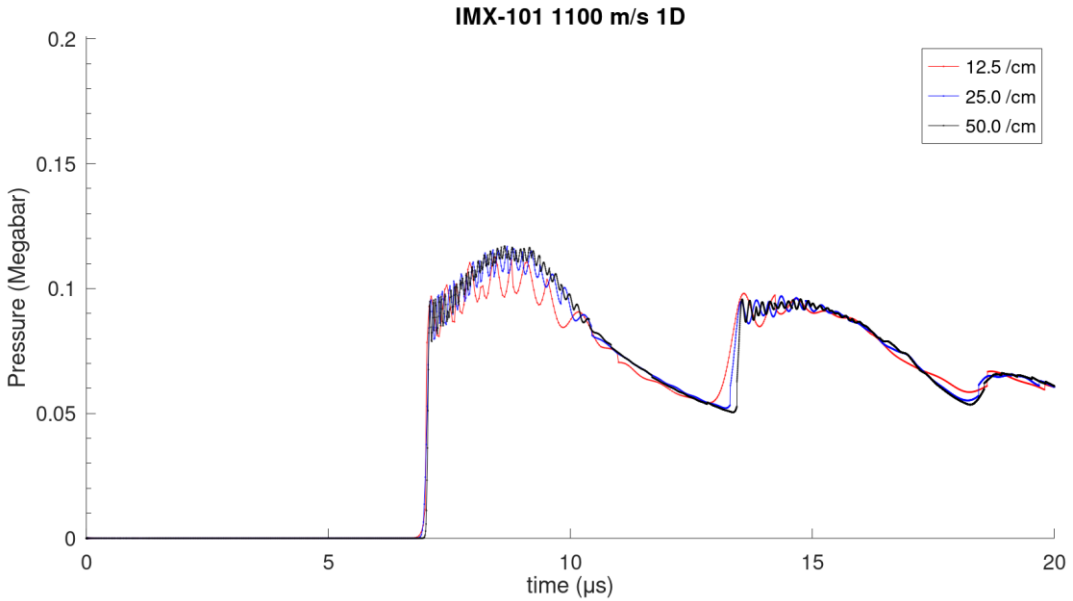
The tracer indicates that TNT is detonating at the time the reaction passes through it.

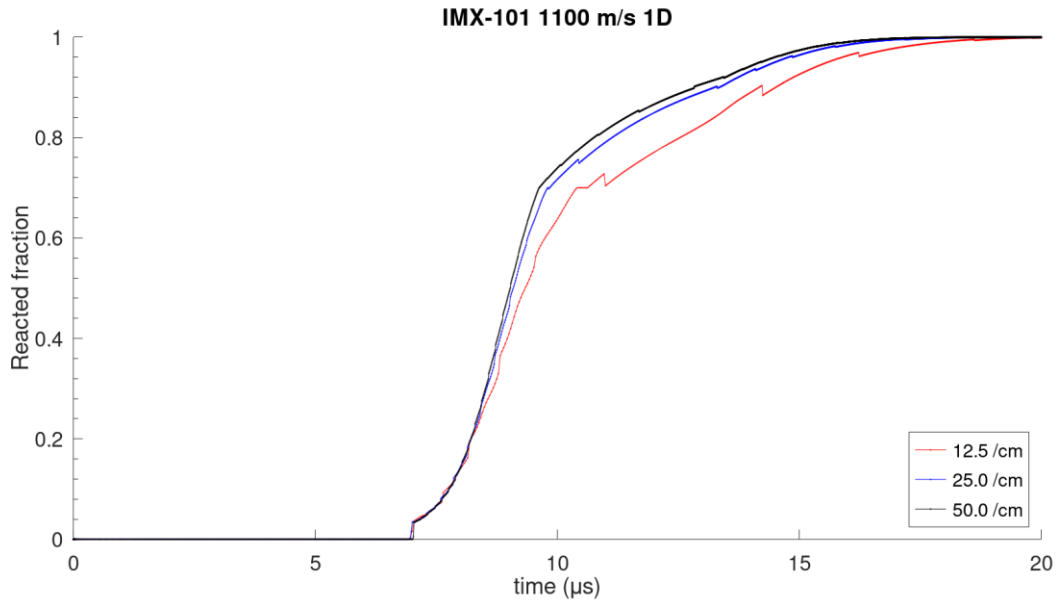
At 12.5 elements/cm, the magnitude of the shock wave is not well captured. The time of arrival of the shock wave differs by approximately 50 nanoseconds. At a resolution of 25 elements/cm the solution is much improved.

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ALE3D 1D RESULTS IMX-101: NEAR GO/NO-GO LIMIT





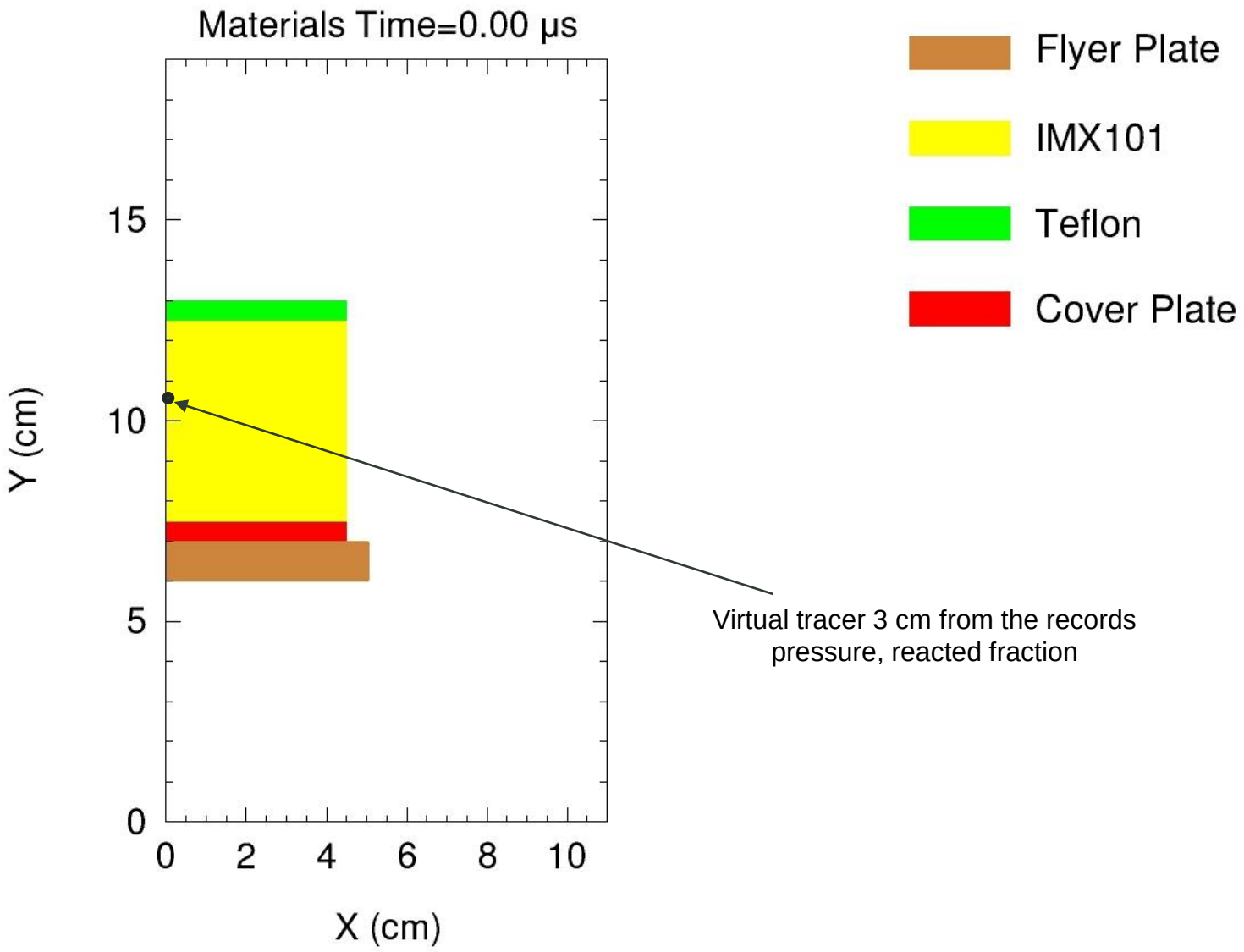
ALE3D results for IMX-101 with a flyer plate velocity of 1100 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

This relatively weak stimulus results in a slow but complete reaction. At 12.5 elements/cm the solution is clearly under resolved. At 25 elements/cm the solution is much improved as it approaches mesh independence.

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2D FLYER PLATE SETUP CTH

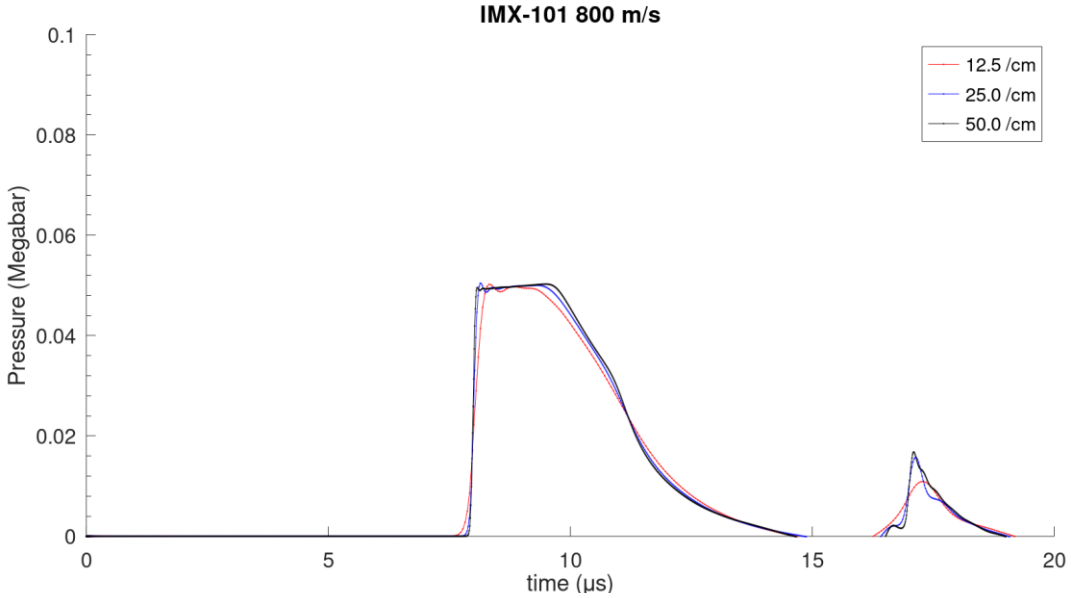
- Flyer plate, cover plate are 4340 steel
- HE is TNT or IMX-101
- Axisymmetric @ x=0 boundary
- Outflow on all other boundaries
- Flyer plate is prescribed an initial velocity [800-1800m/s]
- Pressure and reacted fraction are traced in the HE.
- Three mesh resolutions: 12.5, 25, 50 elements/cm

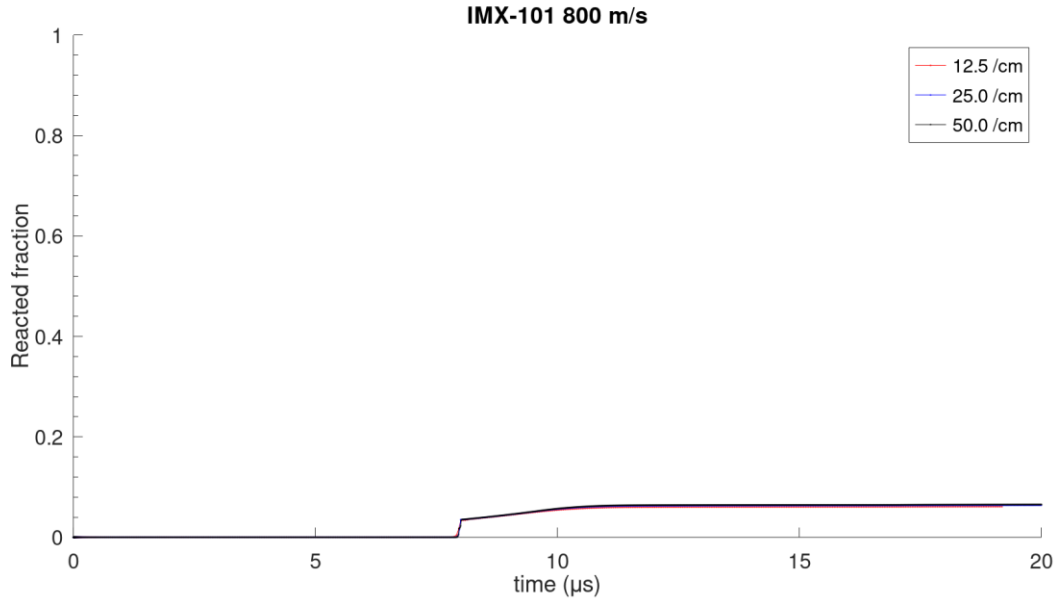


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CTH 2D RESULTS IMX-101: 800 M/S IMPACT VELOCITY





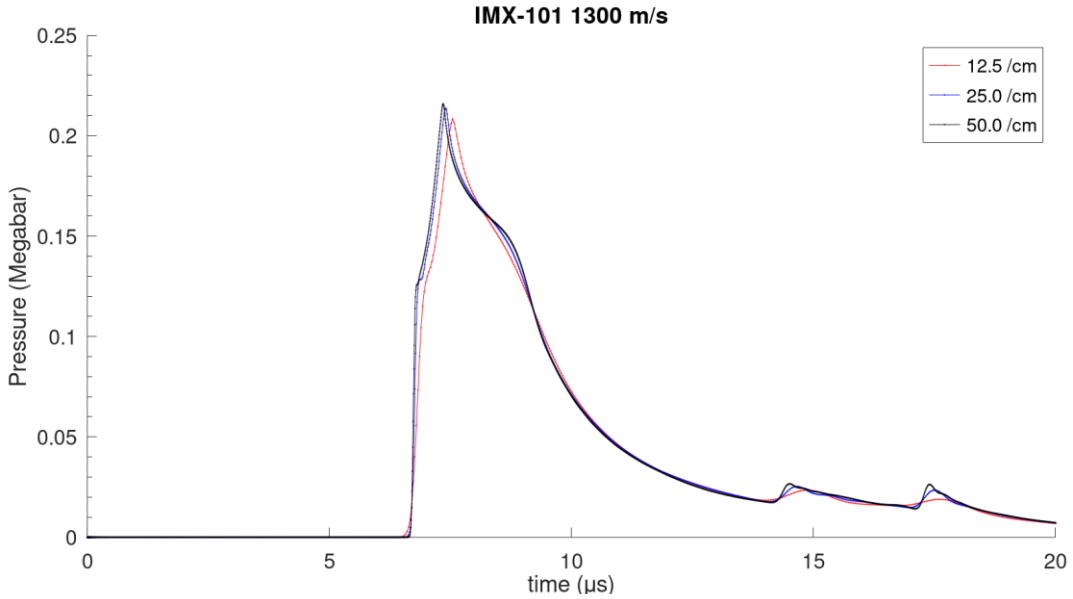
CTH results for IMX-101 with a flyer plate velocity of 800 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

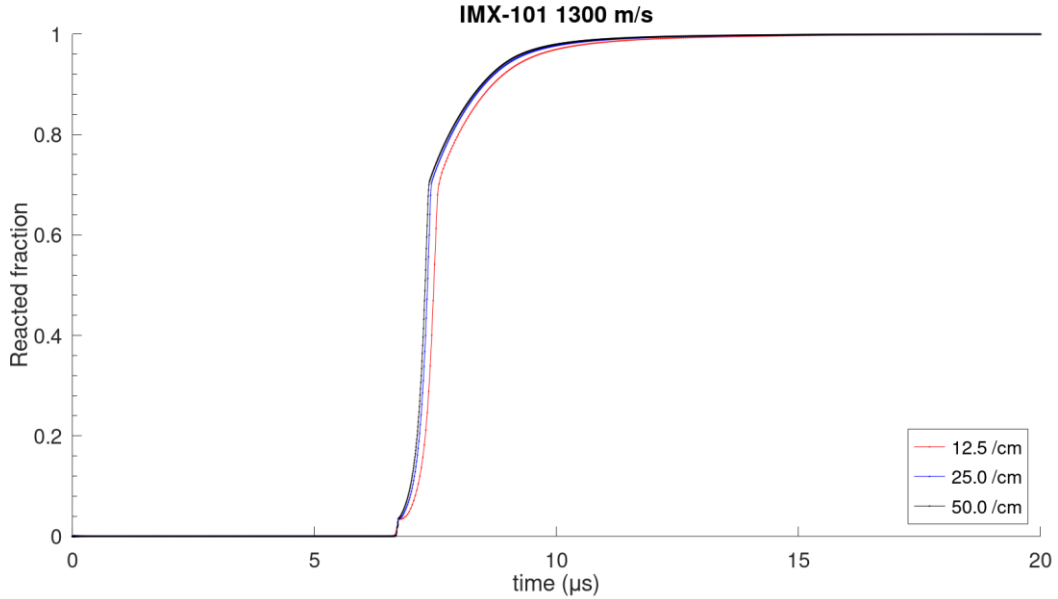
The extent of reaction is small with this flyer plate velocity.

The solution at 25 elements/cm is very close to the solution at 50 elements/cm.

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CTH 2D RESULTS IMX-101: 1300 M/S IMPACT VELOCITY





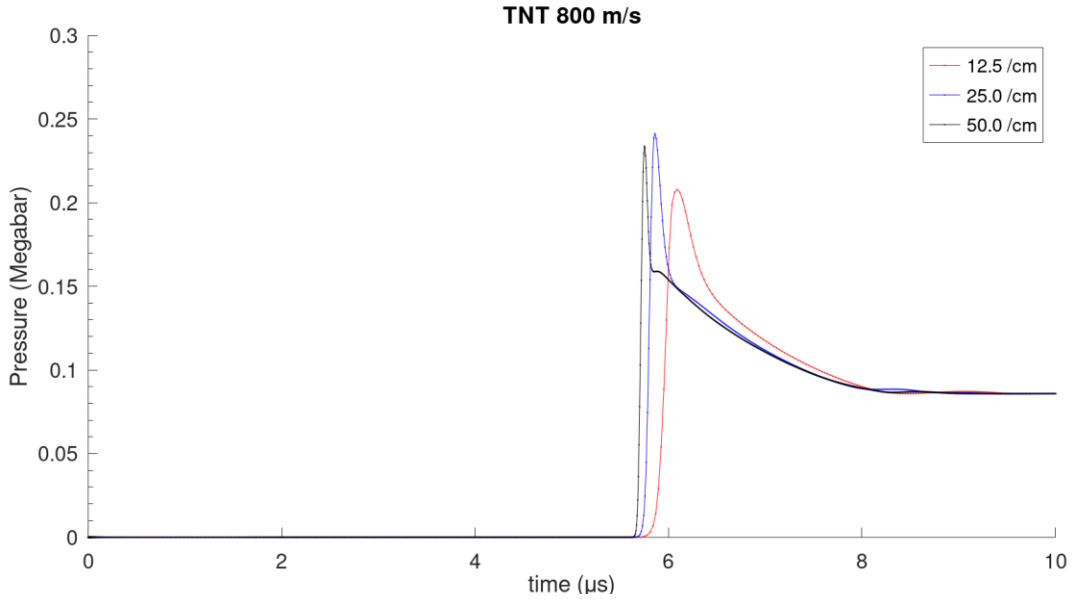
CTH results for IMX-101 with a flyer plate velocity of 1300 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

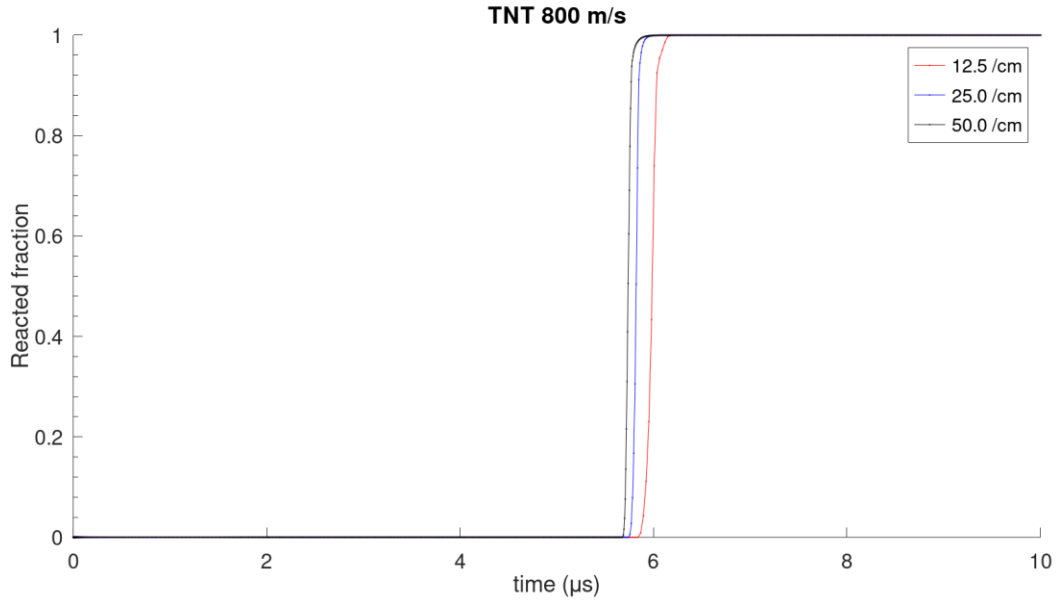
The tracer indicates that IMX-101 is detonating at the time the reaction passes through it.

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CTH 2D RESULTS TNT: 800 M/S IMPACT VELOCITY





CTH results for TNT with a flyer plate velocity of 800 m/s. A virtual tracer records pressure and reacted fraction at a location 3 cm from the leading edge of the initial shock.

The tracer indicates that IMX-101 is detonating at the time the reaction passes through it.

At 12.5 elements/cm, the magnitude of the shock wave is not well captured. The time of arrival of the shock wave differs by approximately 100 nanoseconds. At 25 elements/cm the solution is much closer to mesh independence.

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CONCLUSIONS

- **Reactive flow modeling literature recommends using very high mesh resolutions for I&G likely because:**
  - Model development and validation requires very low relative error.
  - Differences between hydrocodes can affect solution mesh dependence.

- **I&G can provide useful information quickly in some instances using lower-than-recommended mesh resolutions**
  - Very high resolution may not be necessary for certain applied problems.
- **A mesh resolution of approximately 25 elements/cm was determined to provide a good tradeoff between mesh economy and solution accuracy.**

- **Solutions obtained using CTH and ALE3D agreed very well with each other.**
  - CTH is probably the better approach for this problem because its 3D adaptive mesh refinement capabilities allow faster problem generation.

- **Questions?**
  - erik.tolmachoff@cssquaredllc.com

ACKNOWLEDGEMENTS

- **This work was funded by the following Prime Contract/Subcontract:**
  - Prime Contract No. W911NF-15-D-0014 Task/Delivery Order No. W911NF18F0061 from the US Army Contacting Command
  - Leidos Subcontract No. P010220377
- **The presenter would like to acknowledge the support and guidance of the following individuals:**
  - Mr. Anthony Di Stasio, Deputy Director, DPA Title III Program, Industrial Policy, OUSD (A&S)
  - Dr. Kimberly Spangler, Program Manager, Joint DoD/DOE Munitions Program OUSD (R&E)
  - Dr. Paritosh Dave, Senior Program Manager, Leidos

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BACKUP SLIDES

I&G MODEL

$$\frac{dF}{dT} = \underbrace{I(1-F)^b\left(\frac{\rho}{\rho_0}-1-a\right)^x}_{\text{Ignition}} + \underbrace{G_1(1-F)^c F^d p^y}_{\text{Growth Gas}} + \underbrace{G_2(1-F)^e F^g p^z}_{\text{Growth Solid}}$$

Represents the creation of hot spots from compression of voids

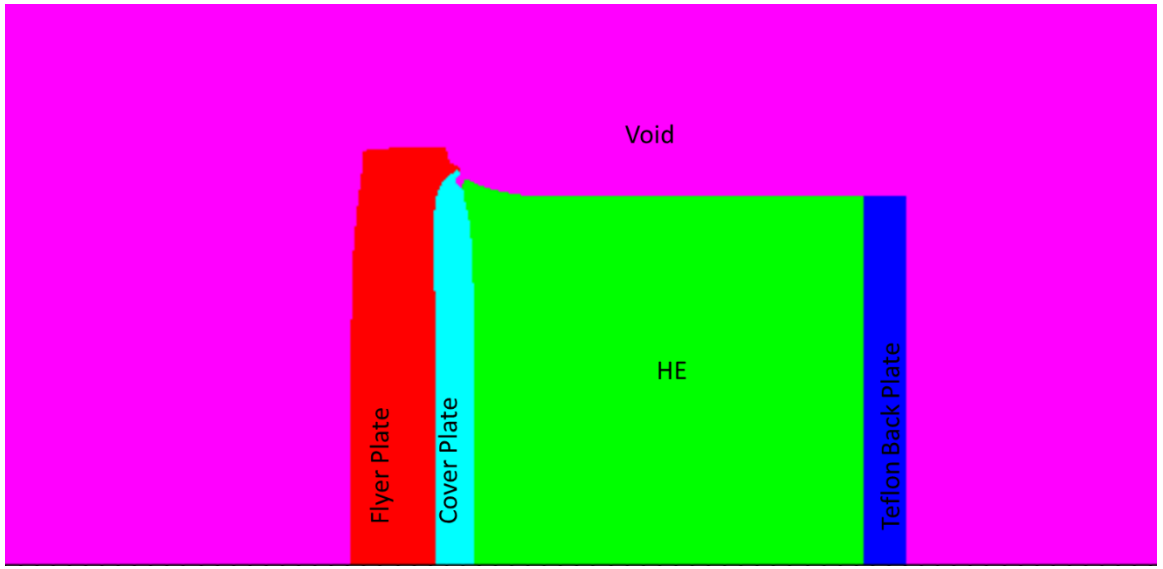
Typically turns off when F reaches ~0.03-0.04

Represents the growth of nascent hot spots, rapid formation of product gasses

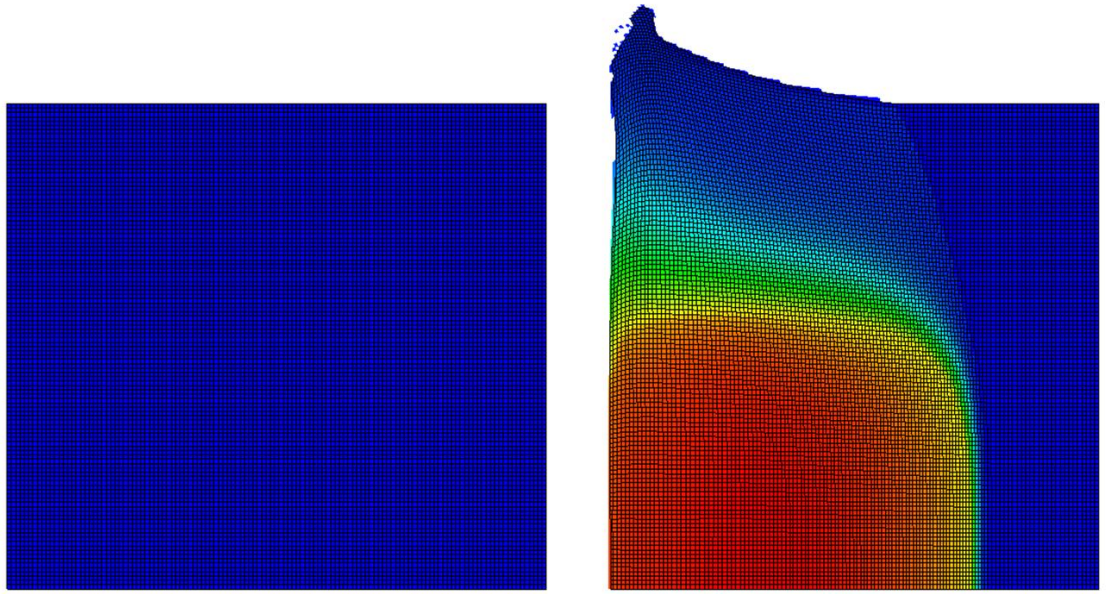
Typically turns off when F >~0.7-0.9

Represents slow formation of solid particles

ALE3D



ALE3D solution of IMX-101, v=1300 m/s at 3.1 μs. Note the distortion of the flyer and cover plates



ALE3D solution of IMX-101, v=1300 m/s at 0 (left) and 8.1 μs (right). The Lagrangian nature of the solution causes slight mesh concentration in the x direction.

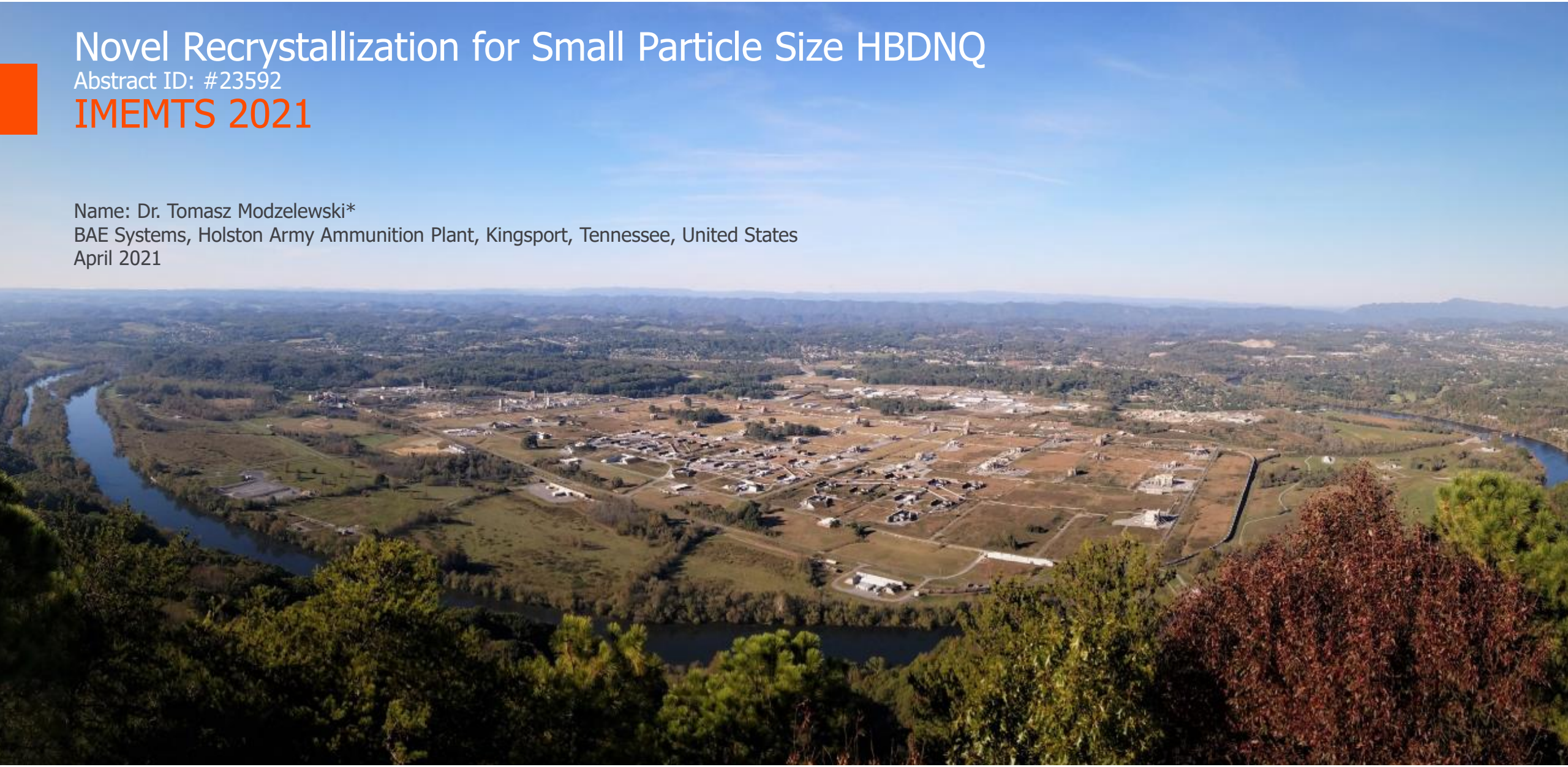
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Novel Recrystallization for Small Particle Size HBDNQ

Abstract ID: #23592

IMEMTS 2021

Name: Dr. Tomasz Modzelewski*
BAE Systems, Holston Army Ammunition Plant, Kingsport, Tennessee, United States
April 2021

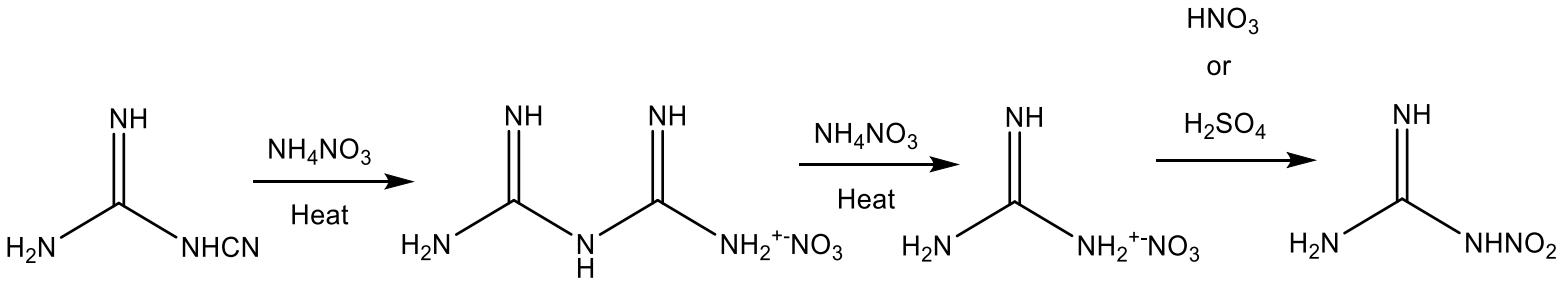


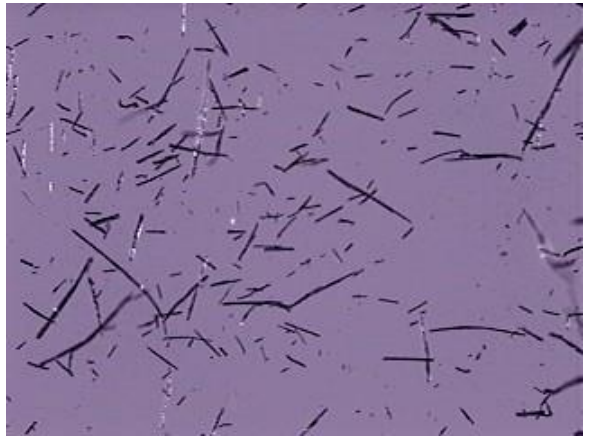
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NQ (Nitroguanidine)

- NQ has long history of use in propellants (ca. 1930s)
  - Simple synthesis from 2-Cyanoguanidine
  - Highly insensitive to unplanned stimuli
- Limited use as explosive filler due to low bulk density
  - Bulk density (< 0.1 g/cc)
    - High aspect ratio needle shapes particles
- Development of High Bulk Density NQ (HBDNQ) allowed for incorporation into insensitive munition formulation IMX-101





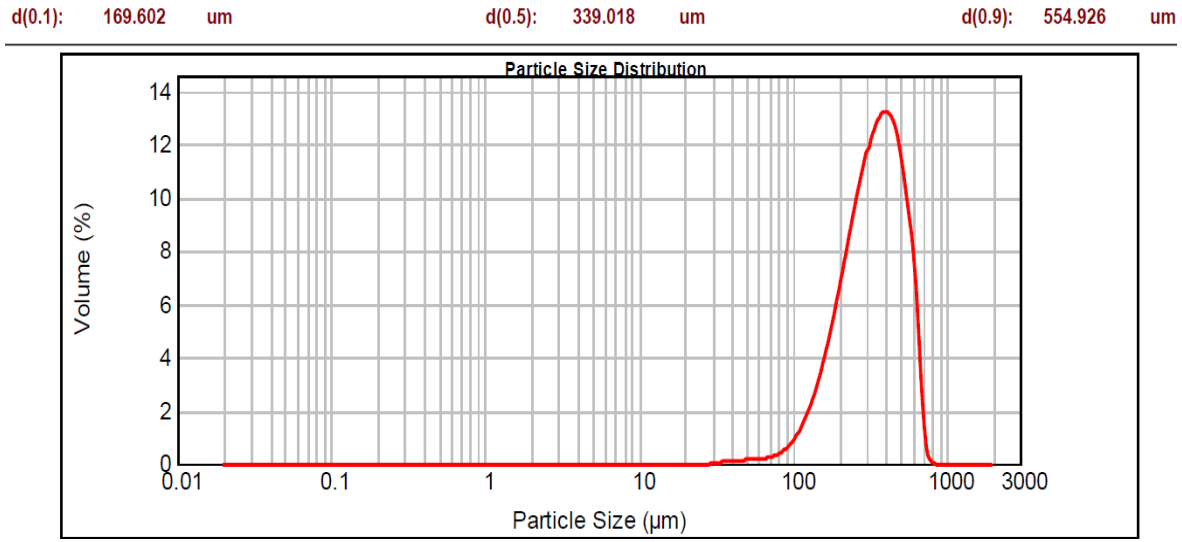
Low Bulk Density NQ

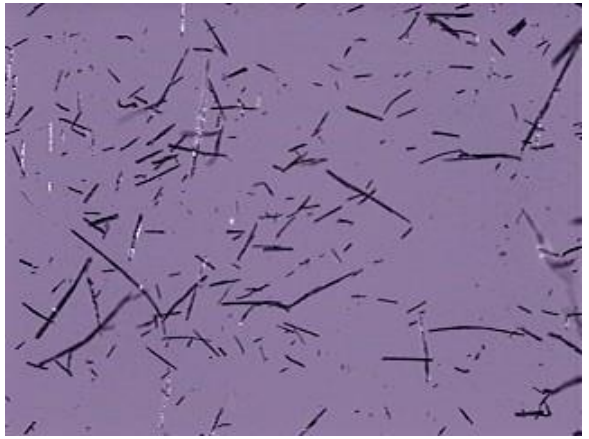
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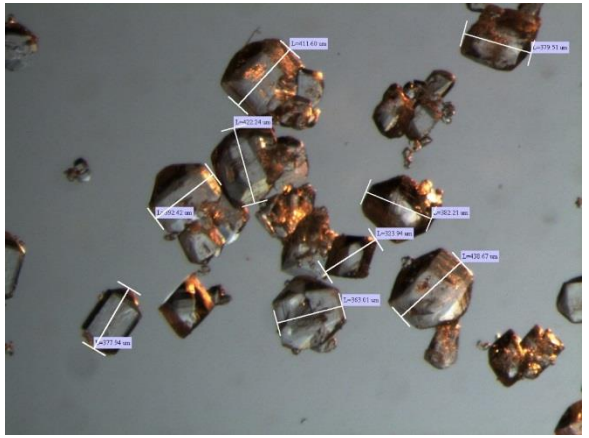
High Bulk Density Nitroguanidine (HBDNQ)

- High Bulk Density NQ (HBDNQ) developed at HSAAP in 2008
  - Dilute acid mediated recrystallization
- Typical particle size
  - d_{10} ~ 100 µm
  - d_{50} ≤ 400 µm
  - d_{90} ≤ 750 µm
- Acidity: ≤ 0.06%
- Purity: ≥ 98%
- Density: ≥ 0.8 g/cc
- Available at full production scale from HSAAP





Low Bulk Density NQ



High Bulk Density NQ

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New NQ Recrystallization Development

- Significant interest exists within DoD for additional classes of NQ for both propellants and explosive formulations
- A novel crystallization process would complement the current recrystallized HBDNQ at HSAAP
  - Simple and scalable procedure
- Decreased particle size could yield a less sensitive product
- BAE has developed a process for a new class of NQ meeting that requirement

	HBDNQ	1182-94	1182-96	1182-115	1182-126	1182-128
d(0.5) (µm)	300 - 400	50.51	31.71	32.22	89.64	82.10

HSAAP HBDNQ

Lab Batches of new NQ

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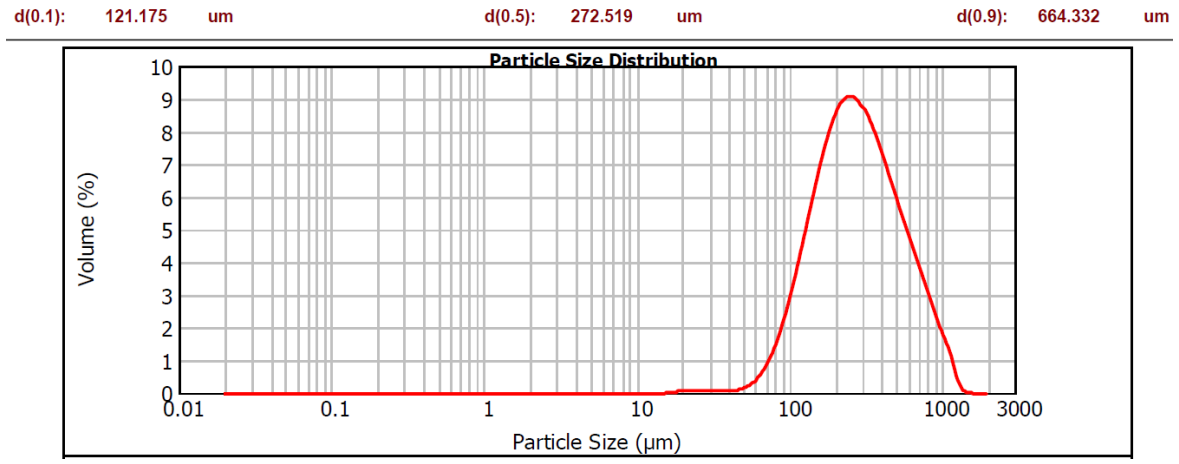
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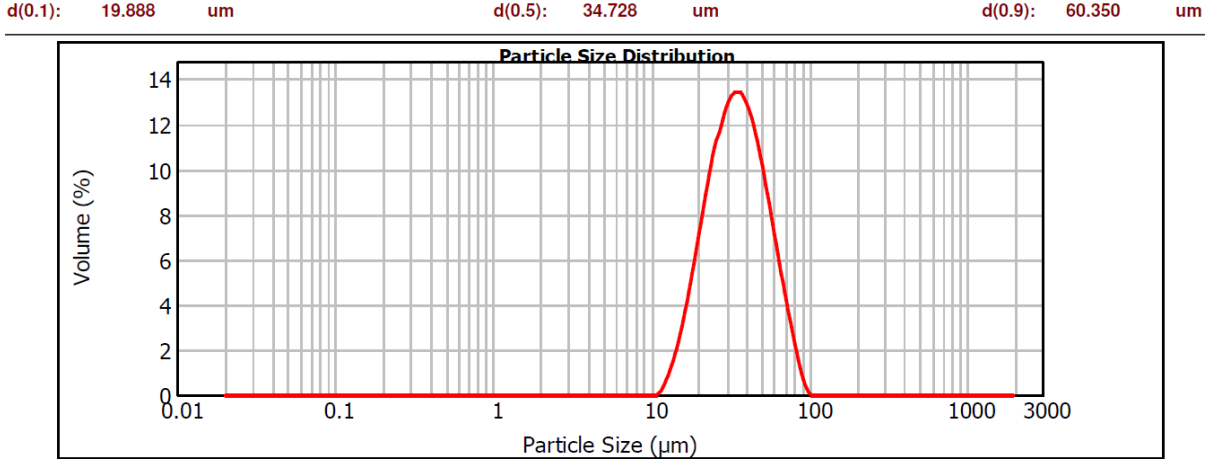
New NQ Recrystallization Development

- Both methods produce a monomodal distribution
- New procedure shows significant decrease in average particle size
  - Narrower distribution compared to standard high bulk density NQ

HSAAP HBDNQ



New ~ 50 µm High Bulk Density NQ

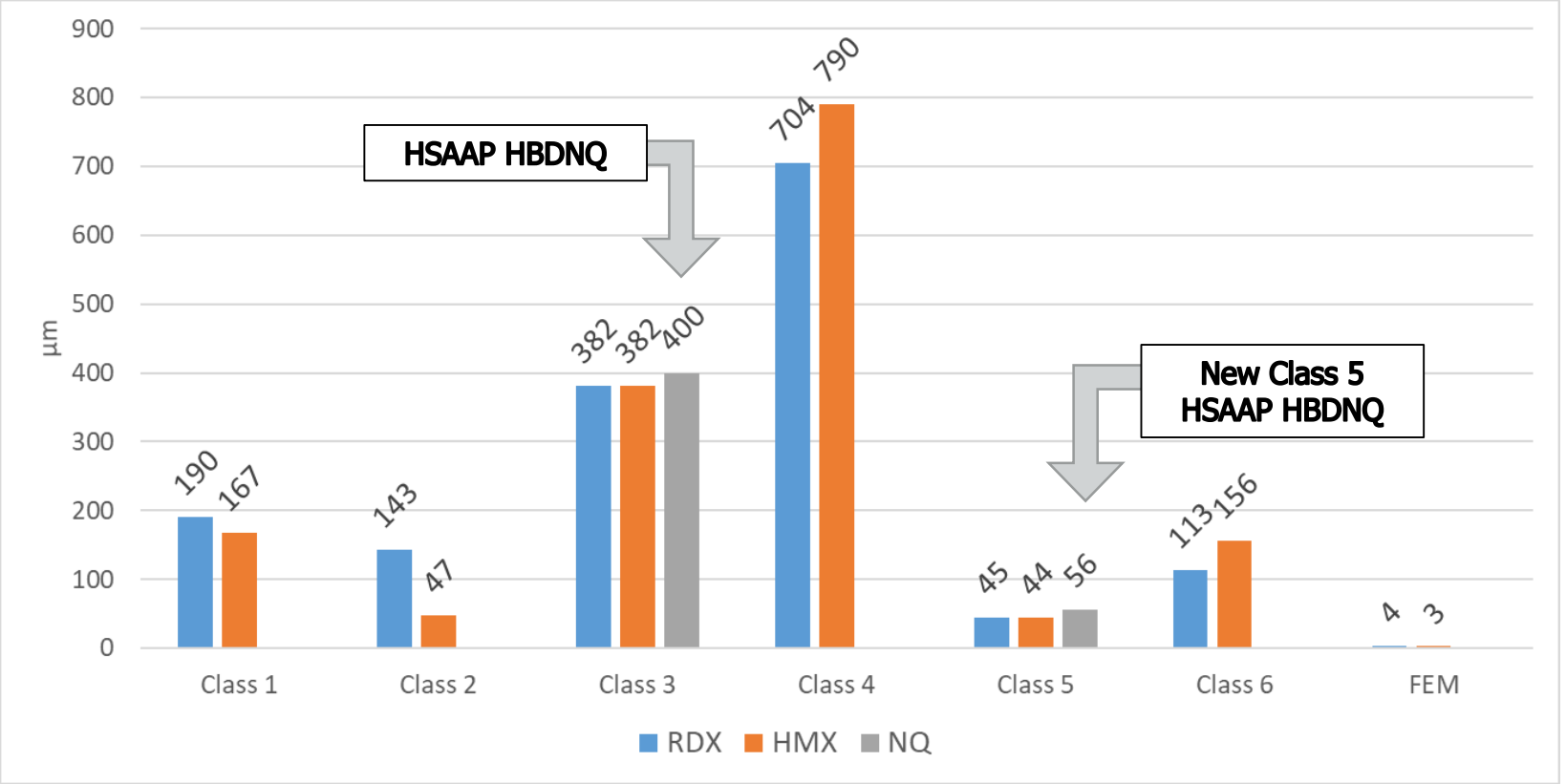


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New NQ Recrystallization Development



- Currently produced HBDNQ similar to Class 3 RDX / HMX
- New ~ 50 µm NQ most similar to Class 5 RDX / HMX

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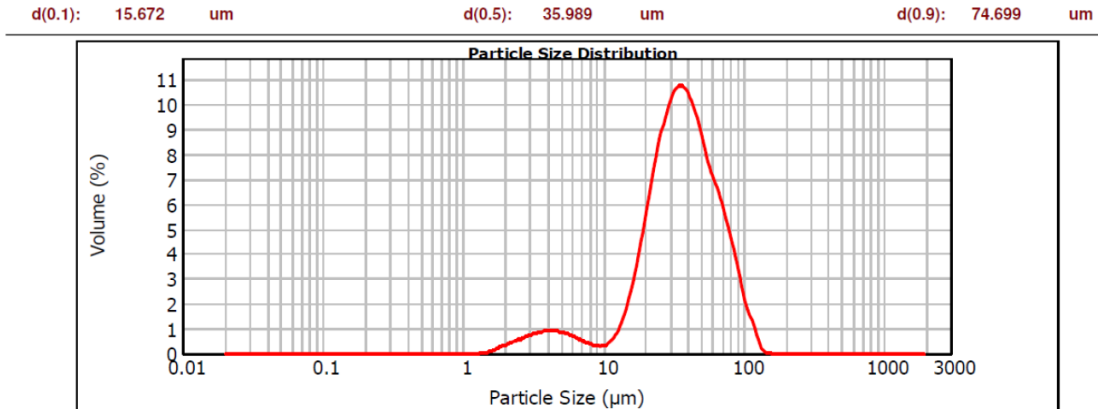
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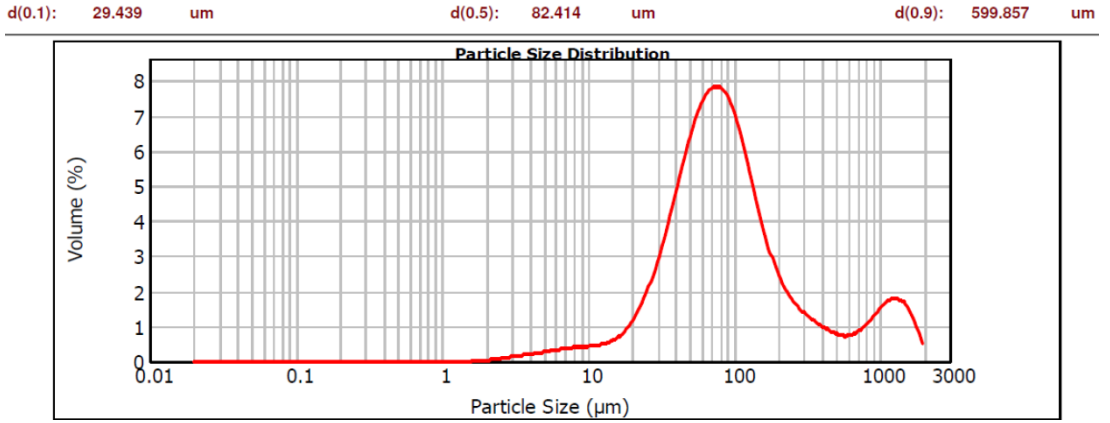
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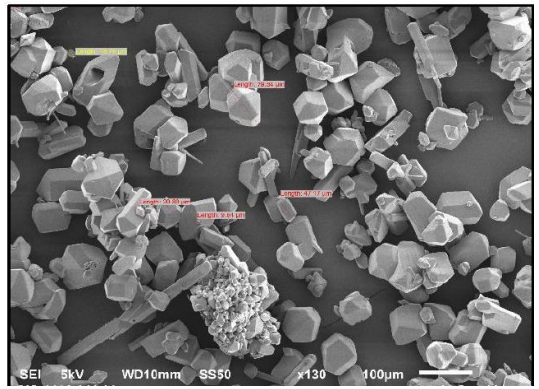
Temperature Control is Key

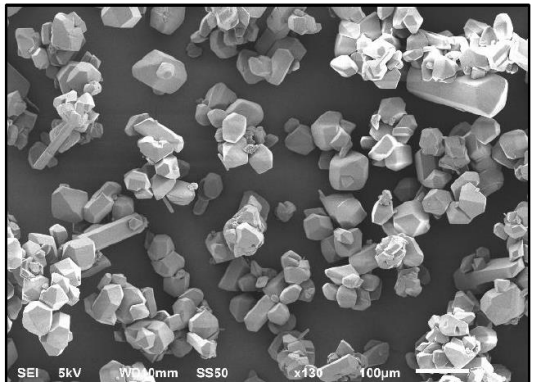
- Initial trials used standard ice bath to chill reaction
  - Increased particle size
  - Bi-modal distribution

- Insufficient surface area / interface between reaction vessel walls and cooling bath
  - Require extreme cooling control to afford sufficient cooling rate to achieve particle size control







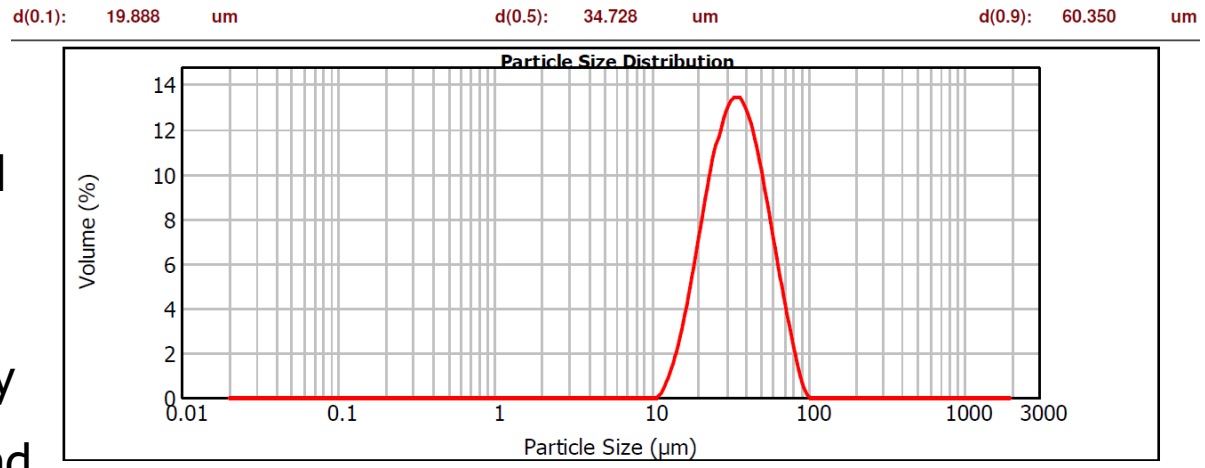


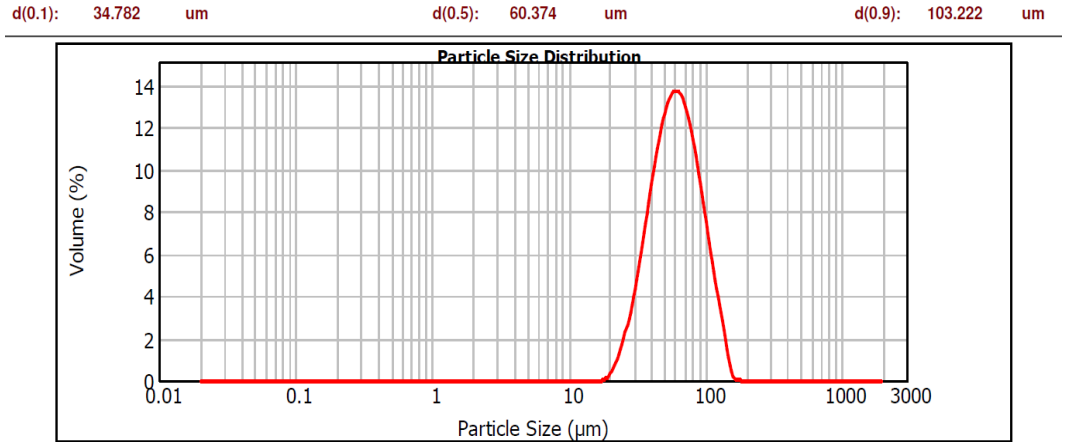
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Temperature Control is Key

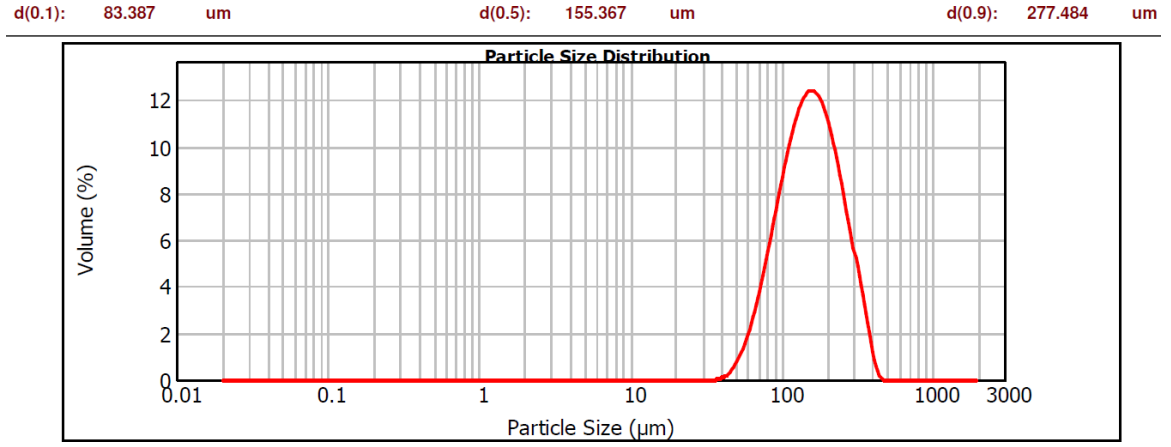
- New procedure requires high degree of temperature control
- At small laboratory scale (up to 1 L scale) this is easily achieved
- When scaling up, ability to remove heat from solution in key
  - Insufficient cooling results in increased particle size and broader distribution

Small scale / good cooling





Large scale / good cooling



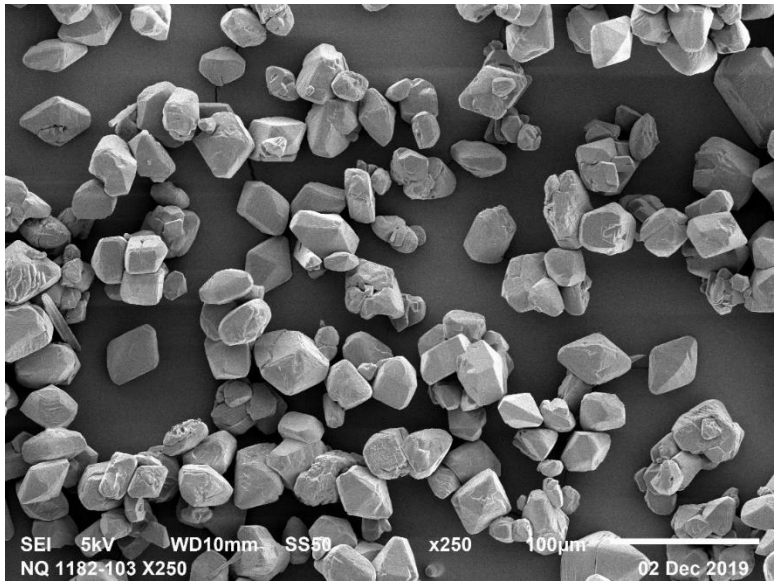
Large scale / insufficient cooling

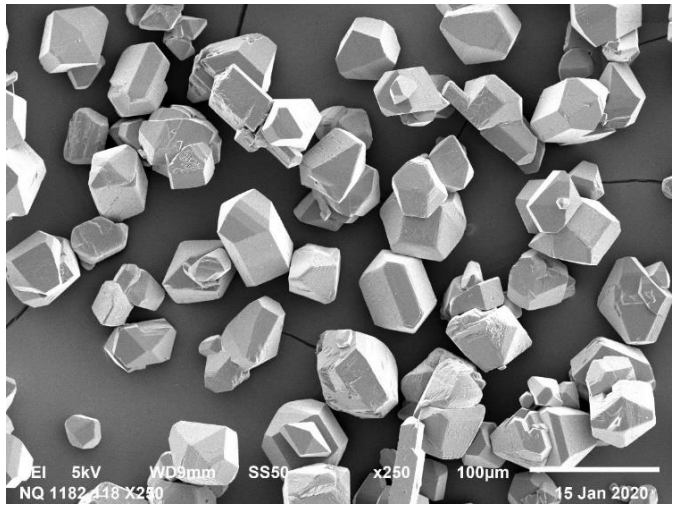
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Particle Shape Analysis

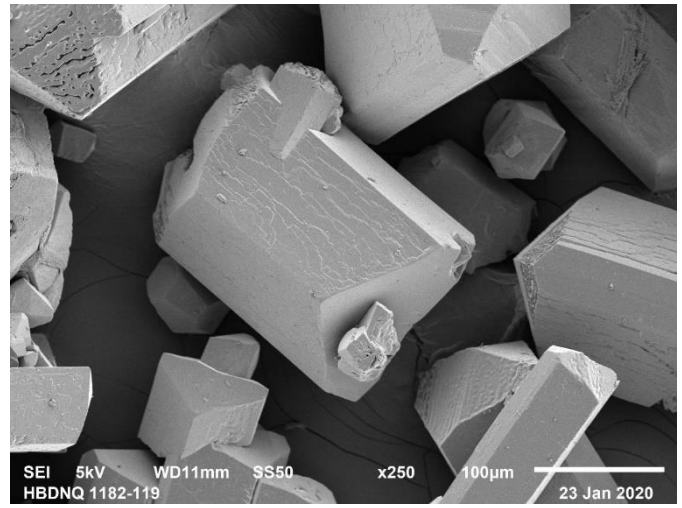
- The crystal shape of the new class of HBDNQ is cubic / bipyramidal
- The smaller scale material is comprised of more uniform shaped particles
- Scaled up material is slightly elongated as compared to smaller scale samples
  - If adequate temperature control is not maintained during entire procedure, resulting particles begin to revert to column shaped elongated particles

Small scale / good cooling





Large scale / good cooling

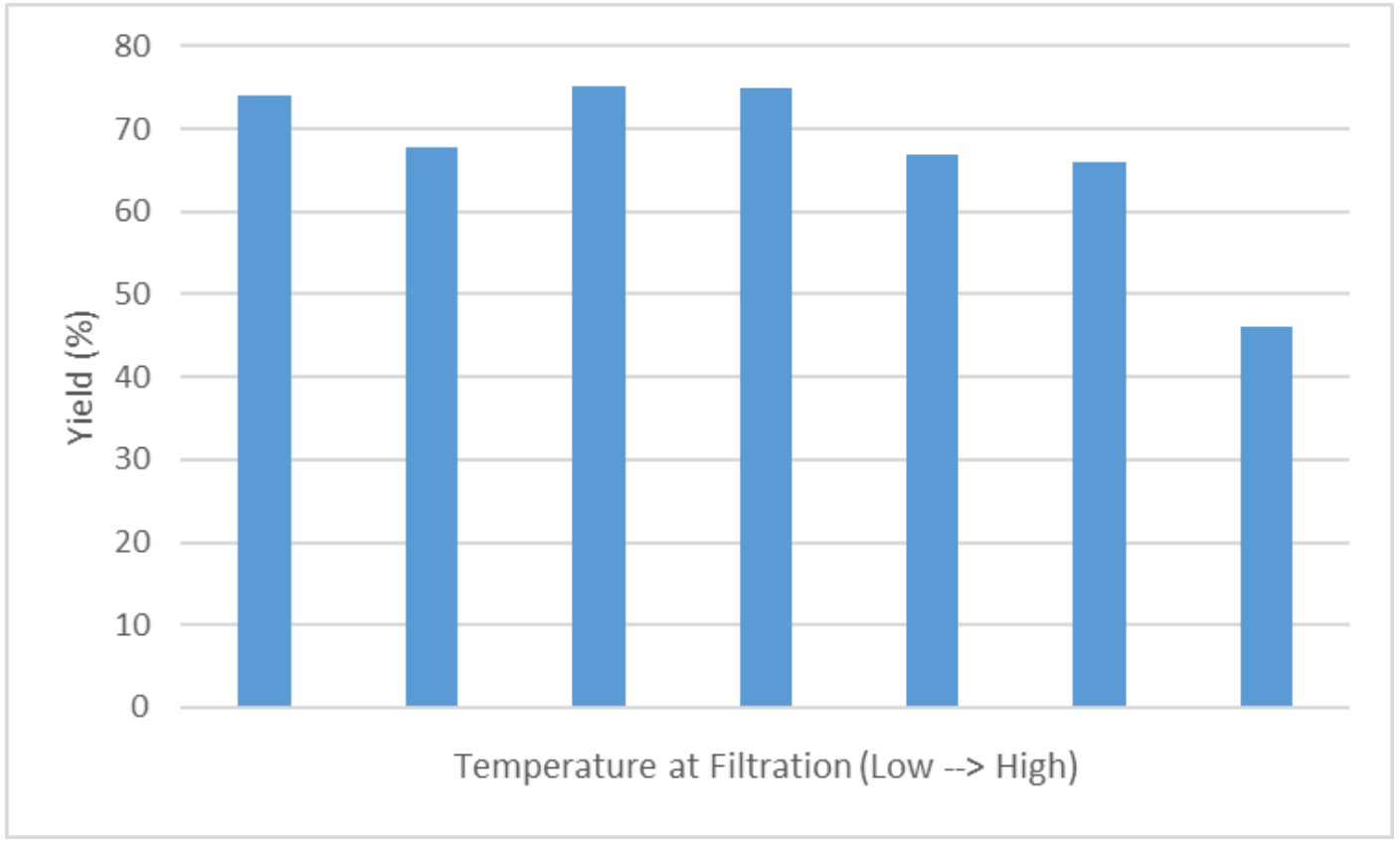


Large scale / insufficient cooling

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Recovery of Final Product

- Total recovery depended on the final temperature of recrystallization solution
- 60 – 75% recovery of NQ when sample is cooled extensively
- Drops to ~ 45% when sample is cooled insufficiently
  - > 30 % drop in recovery



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Path Forward

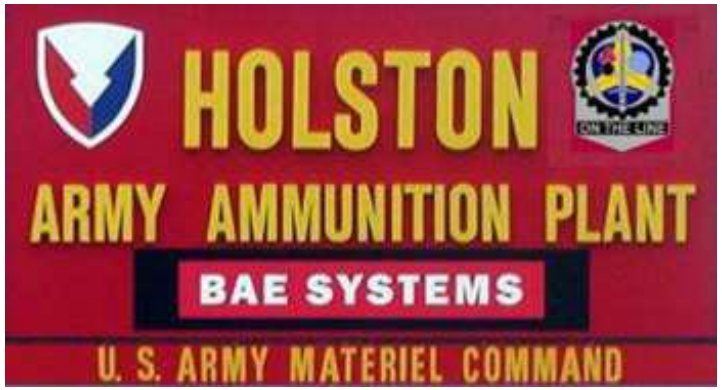
- Transition to pilot plant scale procedure
  - HSAAP D-10 Pilot plant
  - > 100 kg scale
- Determine optimal processing parameters for production scale equipment
  - Additional baffling inside reactor should allow for further reduction in particle size, and lower requirement for highly controlled cooling
- Test new Class 5 NQ in IMX-101 and similar formulation to see effect on viscosity and pourability
- Evaluate Class 5 NQ in:
  - Propellants
  - Pressable and cast-cure formulations

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Conclusions

- A new class of NQ has been developed by BAE Systems at HSAAP
  - Nominal particle size : ~ 50 µm
  - New designation: Class 5
- New material should allow for higher loading of NQ in propellants, and as an explosive filler with a diminished effect on viscosity of formulations





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Acknowledgments

- Matt Hathaway
- Virgil Fung
- Alice Meadows
- Dr. Jeremy Headrick
- Dr. Sarah Headrick
- Dr. Jacob Morris

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MSIAC
Munitions Safety Information Analysis Center

Ministry of Defence

de&s

Supporting Munitions Safety



The influence of mechanical properties on the explosiveness of energetic materials

Stokes Fellowship Project

March-August 2020

Morgan Bolton, DOSG ST1

Kevin Jaansalu, MSIAC, Dr Matthew Andrews DOSG ST1

Morgan.bolton101@mod.gov.uk



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Content

- Purpose of the work
- Material selection
- Defining undamaged materials
- What is damage
- Effect of damage
- Overview for PBX-9501
- Multiple and combined aggressions
- Further work
- Summary

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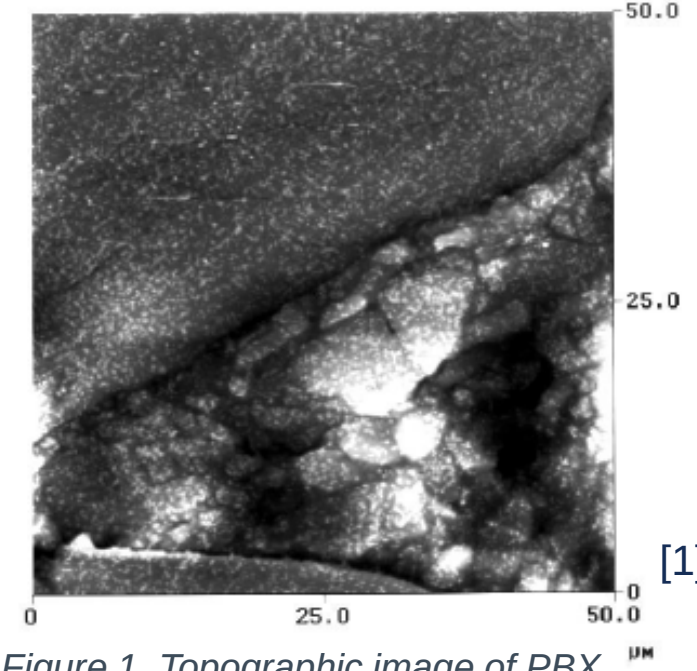
Purpose of this work

- To understand effect of damage on the mechanical properties of an energetic material and on the behavior of the component
- Comparisons between pristine and damaged materials
- Link between material level and component level
  - Material level – change in mechanical properties
  - Component level – change in response to stimuli

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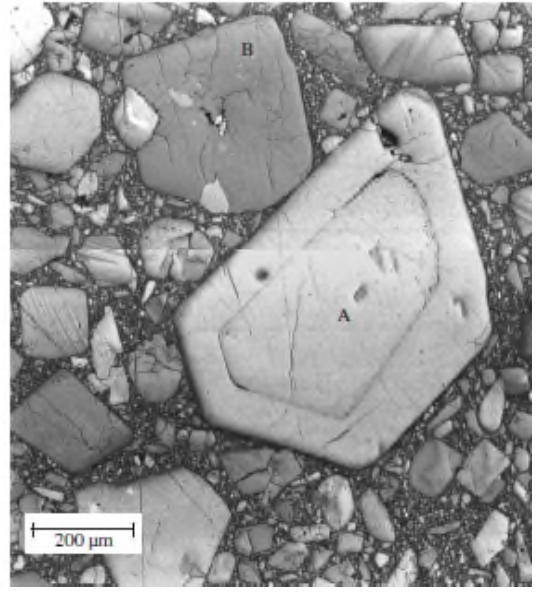
Material selection

- PBX materials
  - Binder changes the mechanical properties of a formulation
  - Wide range of properties
  - Complex
  - Many uses



[1]

Figure 1. Topographic image of PBX



[2]

Figure 2. Optical micrograph of PBX

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Defining undamaged materials

- Lack of clarity and inconsistency with use of terms

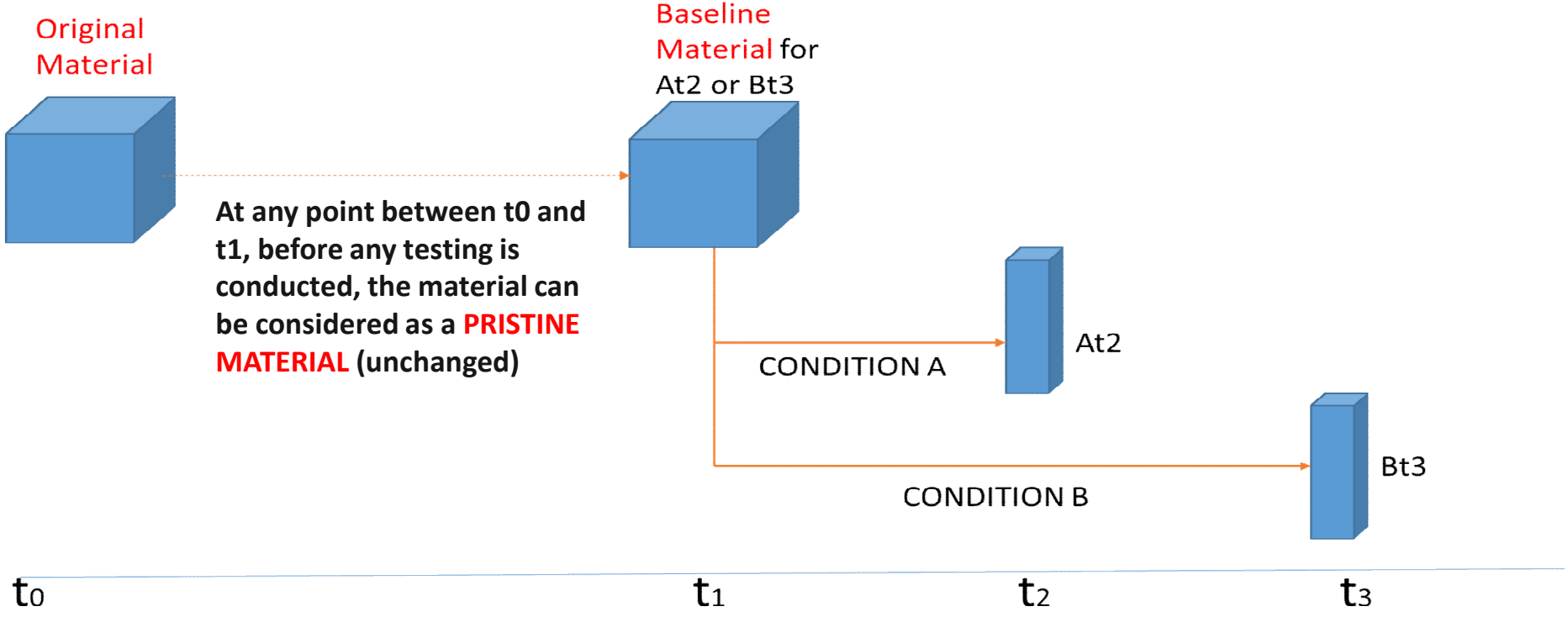
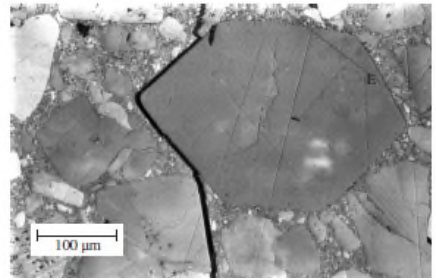


Figure 3. Visual Relationship between the definition for 'original', 'baseline' and 'pristine' materials

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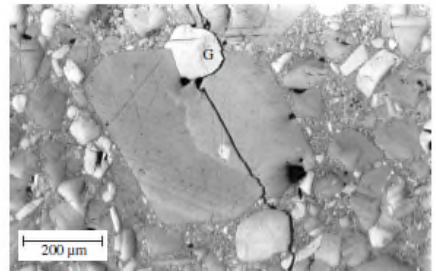
What is damage?

- Change from the original material
  - Visible
  - Measurable
- Can cause a change in performance
- Different sources of damage
  - Mechanical
  - Thermomechanical
- Damage at the material and/or component level
- Cracks, deformation and debonding, porosity and permeability



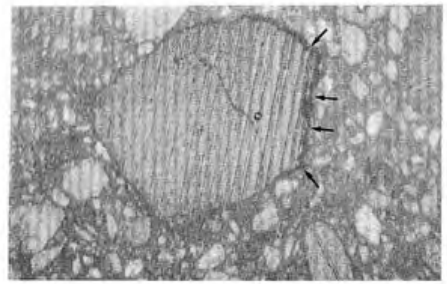
[2]

Figure 4. Interfacial crack



[2]

Figure 5. Internal crystal crack



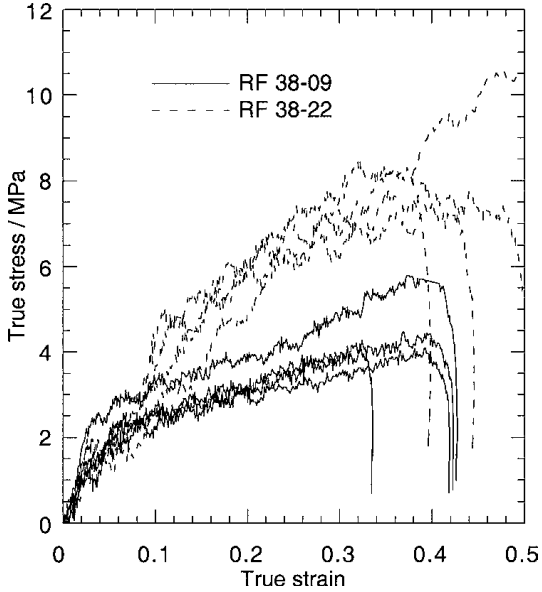
[3]

Figure 6. Micrograph showing debonding

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Factors affecting damage

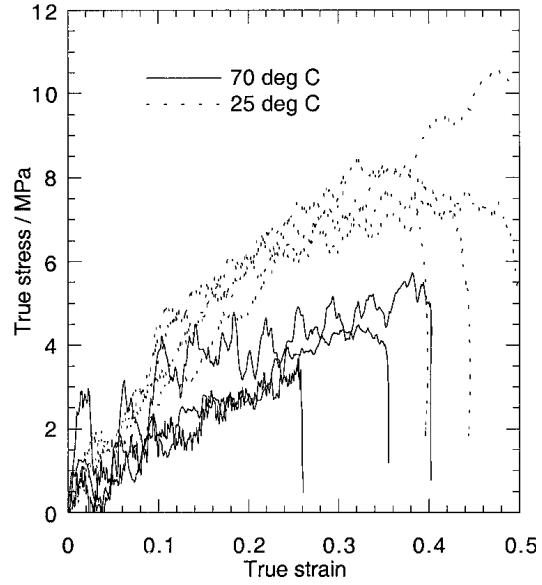
- Material factors
  - Particle size and shape
  - Composition
  - Degree of adhesion



[3]

Figure 7. Stress vs. Strain Graph to show effect of particle size. RF 38-09 RDX 710µm, RF 38-22 RDX 159µm

- Environmental factors
  - Atmospheric pressure
  - Low and high temperatures



[3]

Figure 8. Stress vs. Strain Graph to show effect of temperature

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Microstructural changes

- Visual
  - Longer the temperature insult, the bigger the crack
  - No quantification of amount, location, or distribution of cracks
- Mass and density
  - Greater mass loss at higher temperatures, and longer duration
- Porosity and permeability
  - Increases with temperature and duration of insult
  - Confined vs. unconfined
- Detonation velocity
  - Decreases with increasing temperature

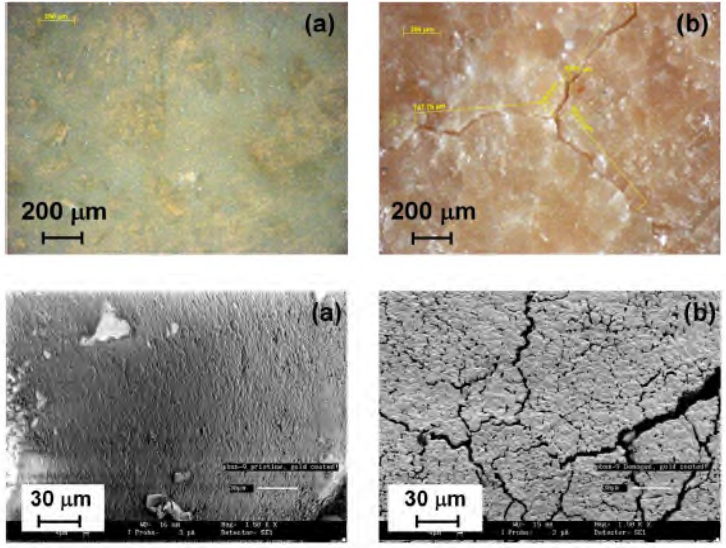


Figure 9. Microscopic images of PBXN-9 before and after damage at 180˚C for 3 hours [8]

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Effects of damage - small scale tests

Table 1. Small scale sensitivity test results for thermally damaged PBX materials

PBX	Explosive Fill	Binder	Condition	Test	Result	Ref
LX-10	HMX 95%	Viton 5%	180 °C 4hr	Impact (Drop Hammer)	Increase 52cm (71cm pristine)	[9]
LX-14	HMX 95.5%	Estane 4.5%	180 °C 4hr	Impact (Drop Hammer)	Increase 75cm (94cm pristine)	[9]
LX-17	TATB 92.5%	Kel-F 800	190 °C 250 °C	Impact (Drop Hammer)	No change (no data)	[10]
PAX-2A	HMX 85%	BDNPA/F 9%	60 °C 12mths	Impact: ERL, type 12, 2.5kg	No change (no data)	[11]
PBX-9501	HMX 95%	Estane 5%	180 °C 4hr	Impact (Drop Hammer)	Increase 82cm (94cm pristine)	[9]
Rowanex-1440	RDX 66%	HTPB 12%	60 °C 12mths	Impact	Increase (no data)	[11]
PBXN-110	HMX 88%	HTPB	70 °C 6mths	Impact	No change	[12]

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Effects of damage - sub scale tests

- Burn area
  - Increases with temperature at which damage is induced
- Burn rate
  - Increases with temperature at which damaged is induced

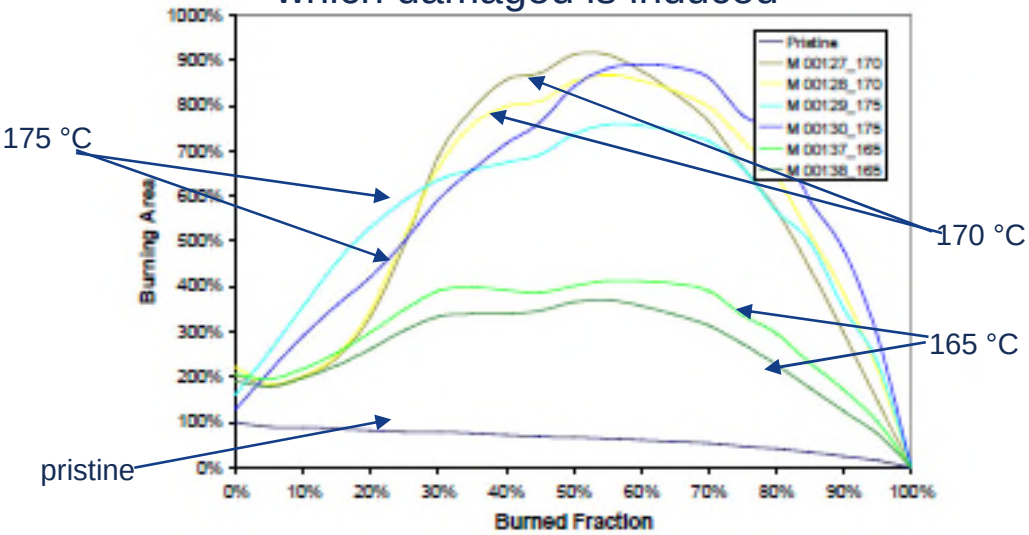


Figure 10. Burn area increase due to thermal damage of HMX-PBX samples [14]

Table 2. Burn rate of PBXN-9 and LX10

Temperature	Burn Rate for PBXN-9 [15] [16]	Burn Rate for LX-10 [9] [10]
90˚C	No change compared to pristine	
110˚C	Only slight acceleration	
150˚C	Approximately 21 times faster than pristine	Slightly faster than pristine
180˚C	2 to 3 times faster when damaged for several hours. 100 times faster	2 to 3 times faster than pristine. 4hours at 180 several orders of magnitude faster
190˚C		Self-ignited

- PBXN-9 – 92% HMX, DOA
- LX10 – 95% HMX, Viton
- HMX-PBX sample 85% HMX, HTPB

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Linking porosity to burn rate

Table 4. Porosity and Burn Rate Results for Thermally Damaged PBX-9501 and LX-10 [17].

Composition	Damage Process	Porosity	Burn Rate
PBX-9501	150˚C for 134 minutes	3.8 %	10^1-10^3 mm/s
PBX-9501	174˚C for 75 minutes	6.5 %	10^4-10^5 mm/s (173˚C)
PBX-9501	174˚C for 227 minutes	6.7 %	
LX-10	150˚C for 134 minutes	3.1 %	-
LX-10	174˚C for 75 minutes	5.2 %	-
LX-10	174˚C for 227 minutes	6.4 %	-
LX-10	180˚C	-	10^3-10^5 mm/s

- LX-10 – HMX 95%, Viton
- PBX-9501 – HMX 95%, Estane
- Porosity increases with temperature and duration for both
- Increased porosity leads to increased burn rate, no clear pattern
- For burn rates the duration of insult was not stated

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Effects of damage - confinement

Table 5. Effect of Confinement on Permeability and Porosity of PBX-9501 [18]

	Temperature and Duration	Permeability, m^2	Porosity
Sample glued to holder, radially confined	180˚C, 4 hours	8.3×10^{-15}	10 %
Unconfined	174˚C, 2 hours	1.4×10^{-13}	>12 %
Sampled glued to holder, radially confined	174˚C, 2 hours	1.4×10^{-15}	6 %

- PBX-9501 – HMX 95%, Estane
- Confinement has a greater effect than temperature and duration on the porosity
- Confinement provides a restraint to the formation of damage

Table 6. Effect of confinement on the Permeability of PBX-9501 [19]

	Permeability, m^2
Confined	2.89×10^{-16} (3 orders of magnitude larger than pristine)
Unconfined	6.88×10^{-14} (5 orders of magnitude larger than pristine)

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Temperature and detonation velocity

Table 7. Table to show the relationship between temperature of insult and detonation velocity [20][21][22]

Temperature	Detonation Velocity of LX-04	Detonation Velocity of HU45 (85% HMX, HTPB binder), km/s
Pristine	8.5	8.3
165˚C	-	7.8
170˚C	-	6.8
175˚C	-	7.0
185˚C	7.7-7.8	

- LX-04 – HMX 85%, Viton
- HU45 – HMX 85%, HTPB
- Temperature causes a decrease in the detonation velocity
- LX-04 has less change over a greater temperature range than HU45
- HU45 detonation velocity increases at 175˚C slightly, suggestion temperature has no greater effect at 170-175˚C?

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Crack size and critical pressure

- Crack size 2-20µm at 180°C, 30 mins
- Crack size 10-100µm at 180°C, 3 hrs
- Could use similar plots to highlight where acceptable limits for damage formation may lie if the acceptable limit for critical pressure is known

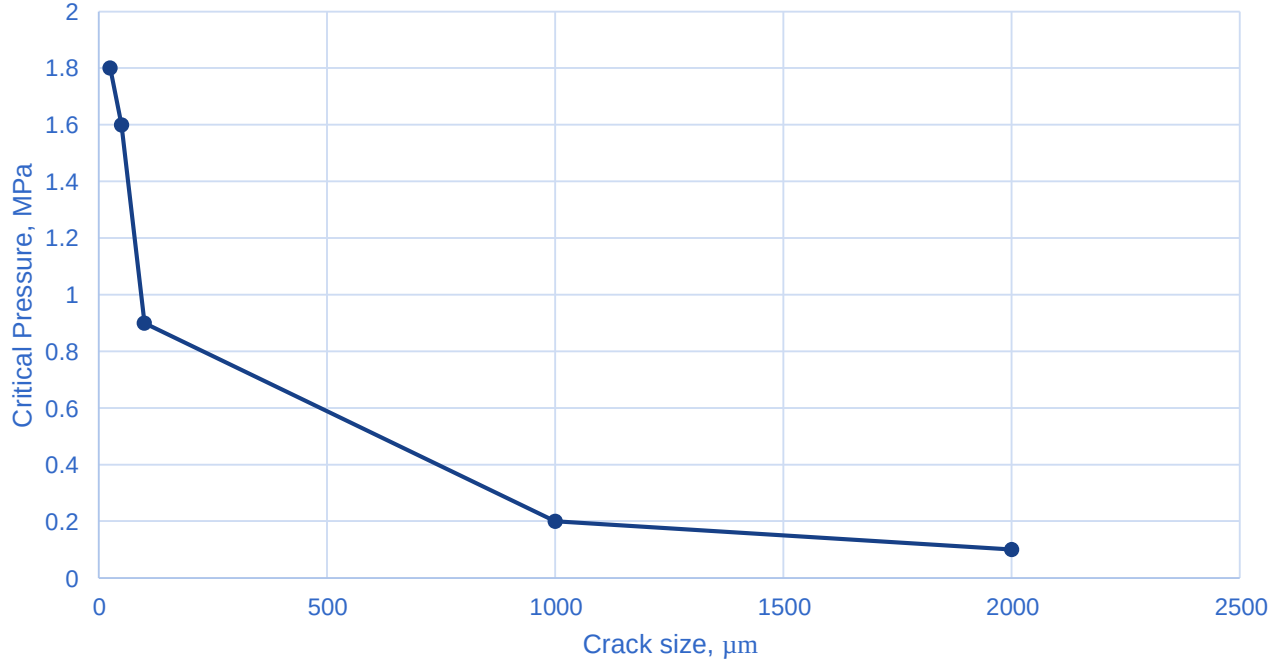


Figure 11. Graph to show the relationship between crack size and critical pressure for PBX-9501

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Overview table for PBX-9501

	Test	Mechanical Damage Response	Thermal Damage Response
Characterisation	Visual	Cracks, 200-300 µm [26] [27] [28]	Cracks, 2-20 µm. pores increase with increasing temperature of insult [26]
	Density		1.53 g/cm^3 (1.81 g/cm^3 pristine) [28]
	Mass		0.7 % decrease [28]
	Permeability		Increased with increasing temperature of insult. Increased with duration of insult [30] [31] [32] [33] [34]
	Porosity		Increased with temperature of insult. Increased with duration of insult [66]
Small-scale Test	Impact test	Changes in sensitivity seen (both increase and decrease) [29]	Increased sensitivity[35][36] [37]
	Friction test		No change [25]
	Spark test		No change [25]
Sub-scale Test	Critical pressure	1.4 ± 0.4 MPa. Increase as crack size decreases [26]	9.2 ± 0.4 MPa. Decreases with porosity and permeability [26] [38] [39] [12]
	DDT tube test		• Porosity increases, run distance decreases • Temperature of insult increases, run distance decreases • Duration of insult increases, run distance decreases [40]
	Burn rate		Increases [25]
	Impact velocity	Decreases [27]	
	Run length to detonation	Decreases as the velocity of impact projectile increases [27]	

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Multiple and combined aggressions

- Multiple bullet impacts
  - US bullet impact tests
  - 10 out of 28 change in response
  - Type V to Type IV
  - Limited details on progression from first impact to second/third
- Slow cook off and a fuel fire
  - Replicate worst case scenario
  - Slow cook off, Type V
  - Fuel fire ignition changed to Type III
- Heat cycling and vibrations
  - Investigation of a Paveway IV accident
  - Unintended burn witnessed in environmental vibration test
  - Cause: combination of prior heat cycling and vibrations



[41]

Figure 12. Photographs of the response of a warhead containing a PBX undergoing a slow cook off followed by a fuel fire. Left to right is the slow cook off, fuel fire ignition and explosion

[41][42][43]

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Summary

- No in-depth damage studies conducted
- Little available data for multiple and combined aggressions but there is evidence of a substantial difference from single aggressions
- Further work
  - Publish experimental detail
  - Realistic damage insults
  - Quantification of damage
  - Links between damage formation and response

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Particle Size Image Analysis of Explosive Formulations & Ingredients ABSTRACT NUMBER

IMEMTS 2021

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04/08/2021

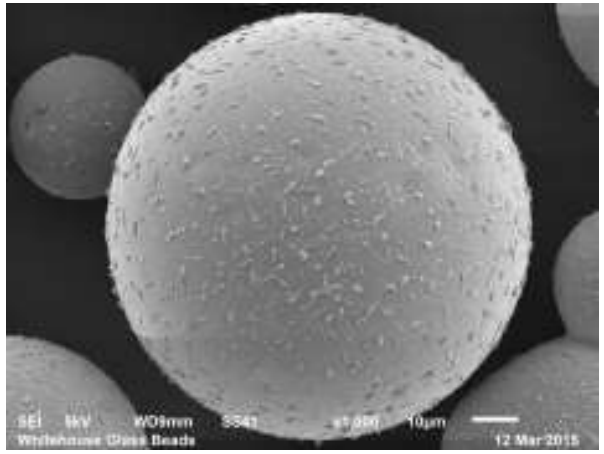


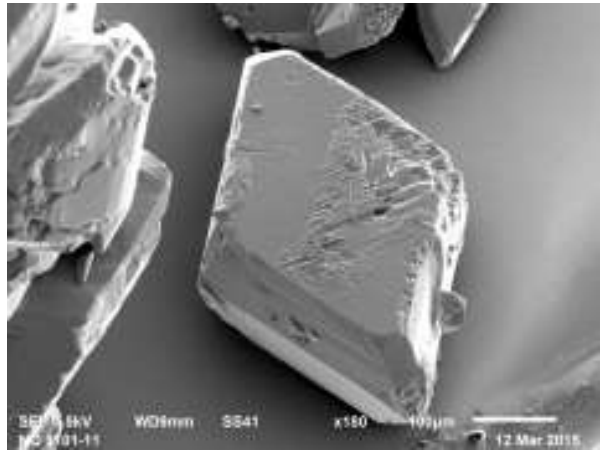
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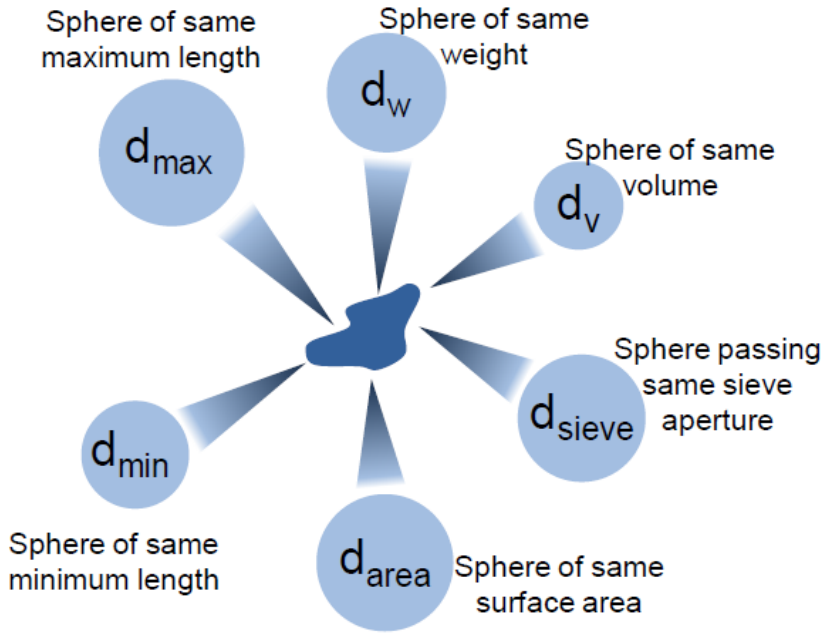
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Particle Size Analysis





- Particles are 3-dimentional objects
- Unless the particles are perfect spheres, they cannot be described by a single dimension, such as radius or diameter
- Most methods define the particle size as an equivalent sphere having a shared property with the actual particle, such as volume, area, or length.
- Different measurement techniques use different equivalent sphere models and therefore will not give the same results for a given particle.



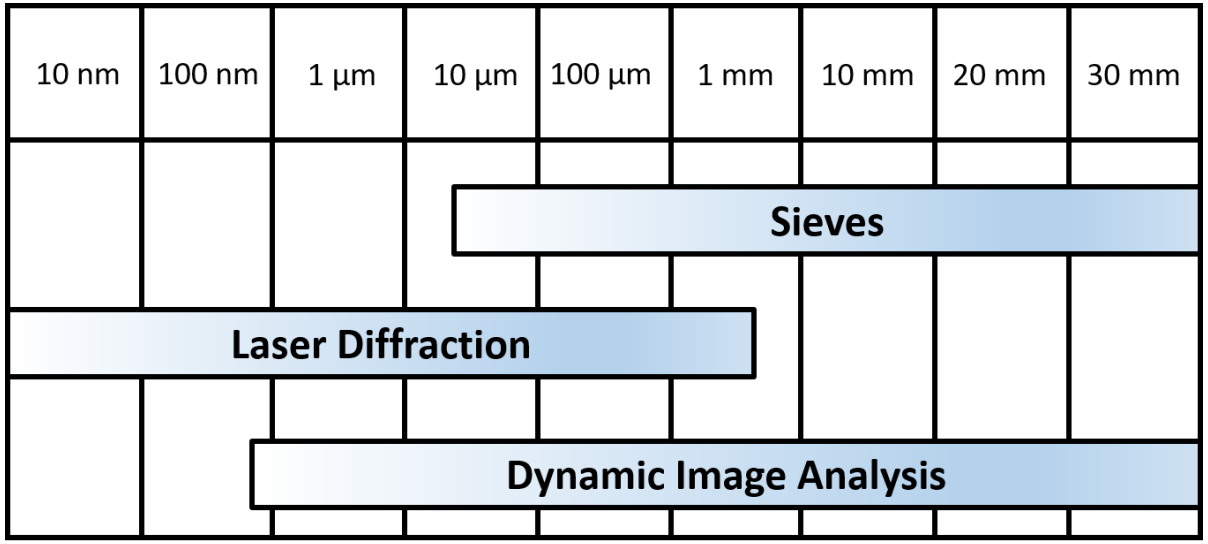
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Particle Imaging Analysis

- Currently most explosives ingredients and granular formulations have particle sizes ranging from approximately 1 micron to 10 millimeters.
- Historically particle size analysis of explosive formulations have been performed by sieving.
- Particle size analysis of explosives ingredients have been done by sieving or laser diffraction methods.
- Particle Imaging Analysis is an alternative technique that can also be used to measure particles in these size ranges.







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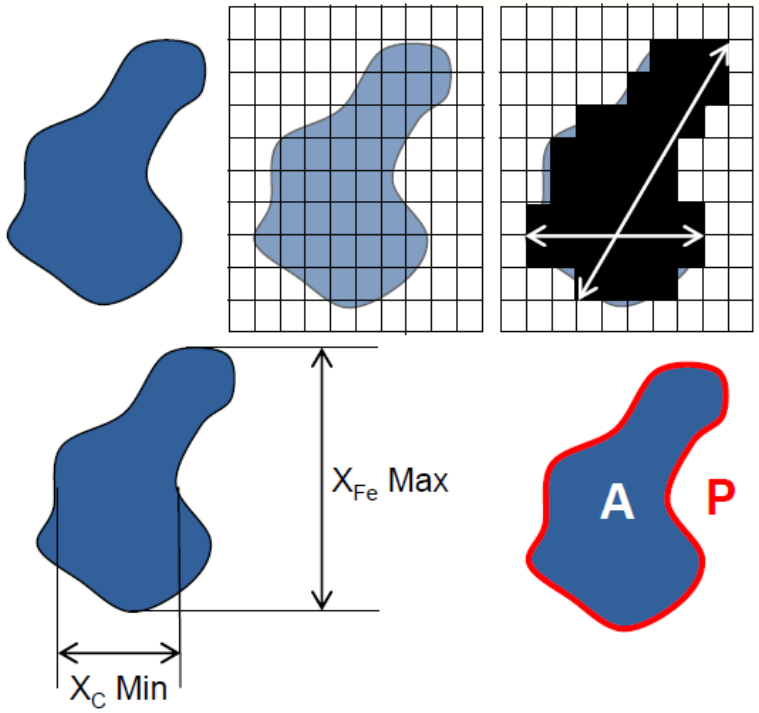
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Particle Imaging Analysis

- Samples are dispersed and passed in front of a digital camera where images are acquired.
  - Static
  - Dynamic
- The images are processed via software to segregate particles from background and to perform image analysis operations on each particle resulting in dimensional measurements.
- These particle measurements can be used to evaluate both size and shape.







$$Aspect\ Ratio = \frac{X_{c\ min}}{X_{Fe\ max}} \qquad Sphericity = \frac{4\pi A}{P^2}$$

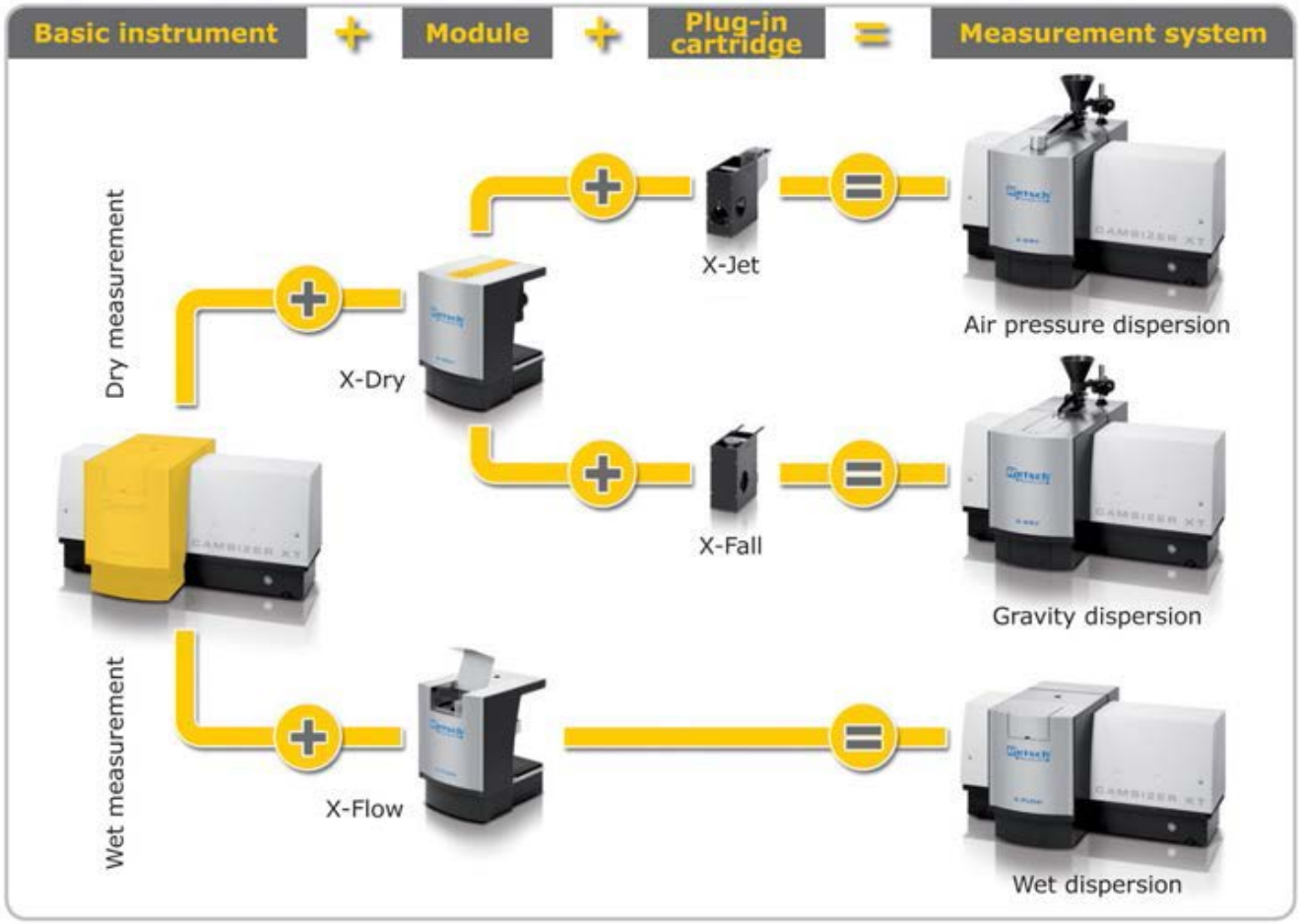
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Particle Imaging Analysis

- Interchangeable module and cartridges makes the system highly adaptable to different sample types.
- X-Dry - X-Jet: Air dispersion
- X-Dry - X-Fall: Gravity dispersion
- X-Flow: Wet dispersion



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Explosive Formulations by Dynamic Image Analysis

- Particles that are dry and free flowing can be analyzed by a vibration feed table and gravity dispersion.
- Materials fall freely past the camera.
- There is no physical means of dispersing agglomerates of particles in the system.





Formulation Methods Developed
Composition A5
CXM-AF-7
LX-14/17
HMX Mains
IMX- 101/104
PBX-9502
PAX-3
PBXN-X (7, 9, 10…)

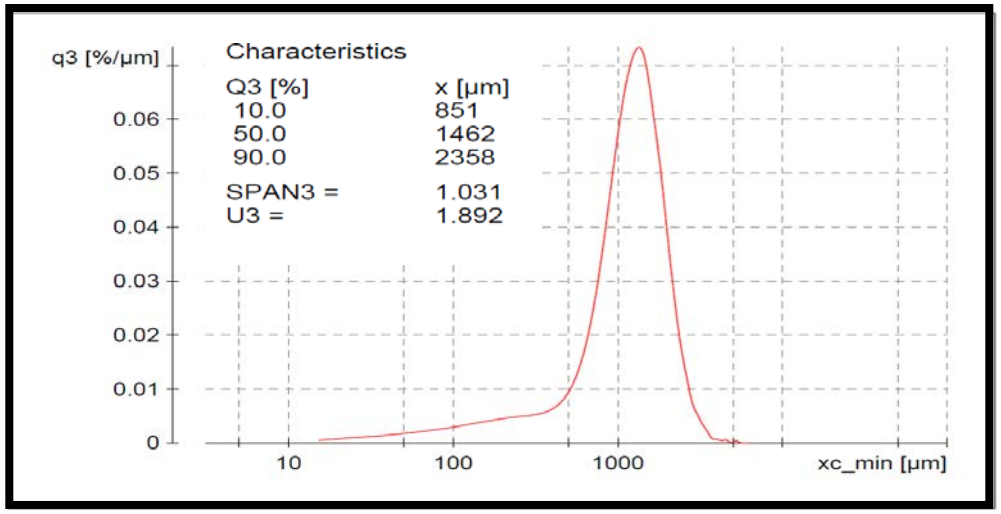
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Explosive Formulations by Particle Image Analysis

- Data
  - % Passing/Retained
  - Shape Parameters
  - Distribution curve
- Dynamic Image Analysis
  - Batch Information Retained
    - Reprocess data in the future
    - Assess Performance Issues or Optimize PSD Parameters



Size class	[µm]	p3 [%]	Q3 [%]	SPHT3
> 4750		0.2	100.0	
2360 -	4750	9.8	99.8	0.894
1700 -	2360	24.7	90.0	0.902
1400 -	1700	19.9	65.3	0.904
1180 -	1400	16.2	45.4	0.901
850 -	1180	19.2	29.2	0.895
180 -	850	9.5	10.0	0.879
0 -	180	0.5	0.5	0.823

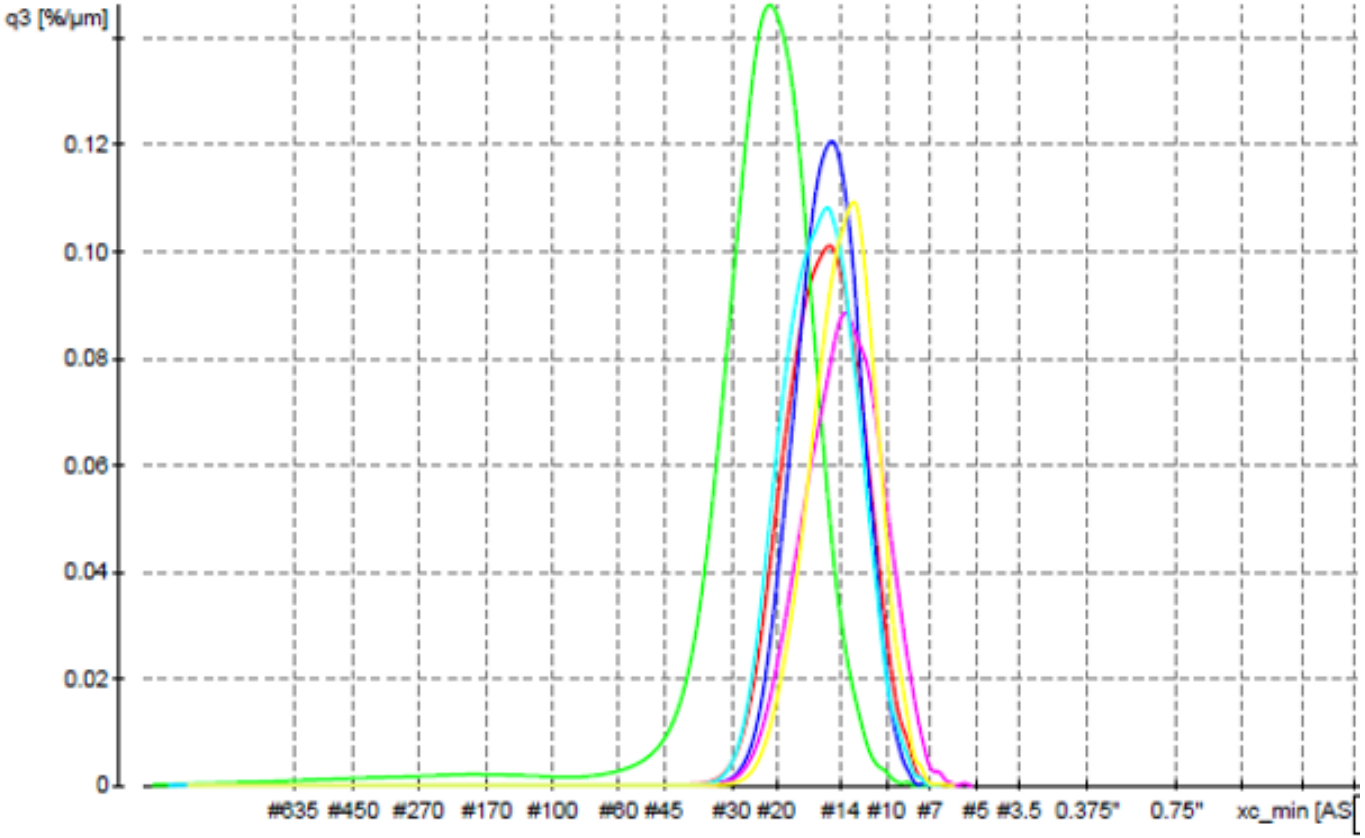
Size class	[µm]	p3 [%]	Q3 [%]	SPHT3
> 3350		1.5	100.0	
2360 -	3350	8.5	98.5	0.894
1700 -	2360	24.7	90.0	0.902
1400 -	1700	19.9	65.3	0.904
1180 -	1400	16.2	45.4	0.901
850 -	1180	19.2	29.2	0.895
710 -	850	4.5	10.0	0.885
600 -	710	2.1	5.5	0.879
425 -	600	1.6	3.4	0.876
180 -	425	1.3	1.8	0.865
45 -	180	0.5	0.5	0.825
	< 45	0.0	0.0	0.761

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Explosive Formulations by Particle Image Analysis



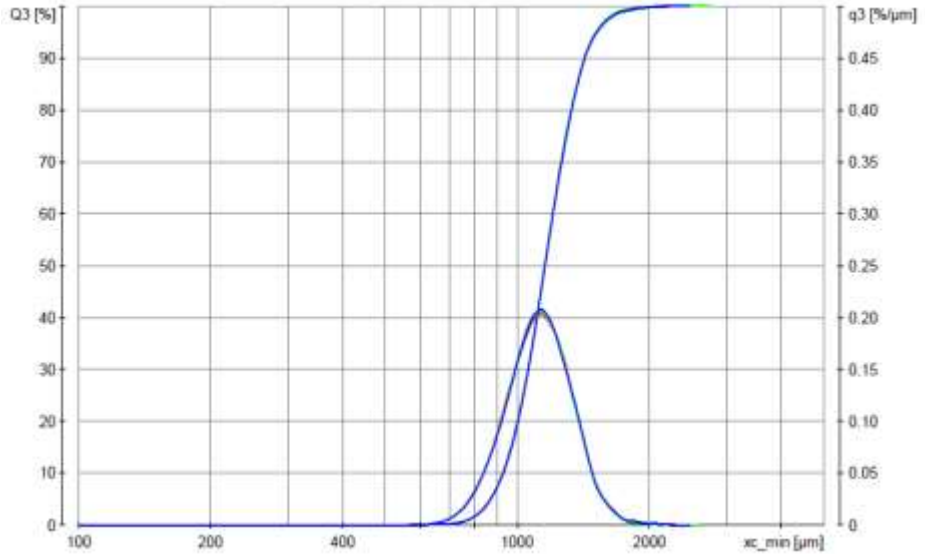
- PBXN-X
  - All data passed MIL-SPEC PSD
  - PSD curve highlights differences.
- Formulation Advantages
  - Full Sieve Range
  - Information collected rapidly
  - Small sample size
  - Distinguishes PSD variation between batches that traditional sieving overlooks.

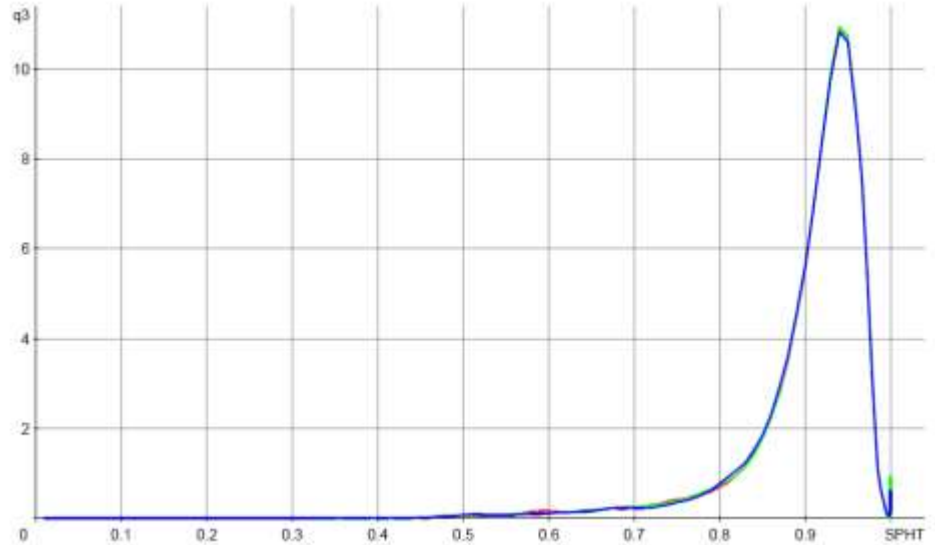
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Explosive Formulations by Particle Image Analysis





Replicate	D10 (µm)	D50 (µm)	D90 (µm)
1	926.1	1156.3	1425.7
2	928.3	1154.8	1422.8
3	928.9	1156.7	1425.7

Replicate	Sphericity	Symmetry	Aspect Ratio
1	0.908	0.928	0.765
2	0.908	0.929	0.766
3	0.908	0.928	0.766

Replicate analysis of formulation products indicate good repeatability for both size and shape analysis

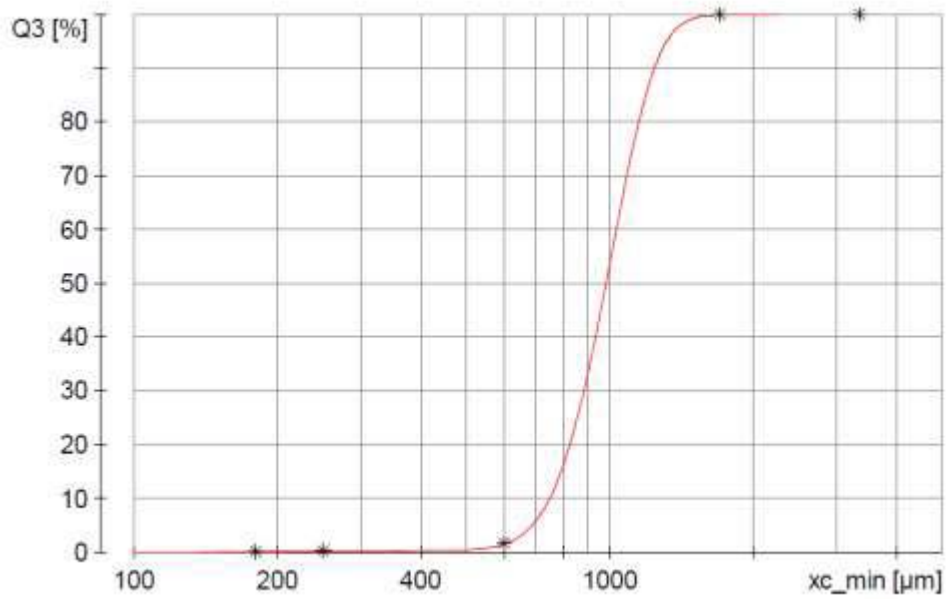
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Explosive Formulations by Particle Image Analysis

- Sample was analyzed by Image Analysis and then by sieving using sieve sizes from product specifications
- Good Agreement to sieving data however data only describes the extremes.



RED – Cumulative Distribution (% passing)

* - Sieving Data

Sieve #	Sieving (% passing)	PIA (% passing)
#6	0.0	0.0
#12	0.0	0.1
#30	98.5	98.4
#60	1.4	1.4
#80	0.0	0.
PAN	0.1	0.1

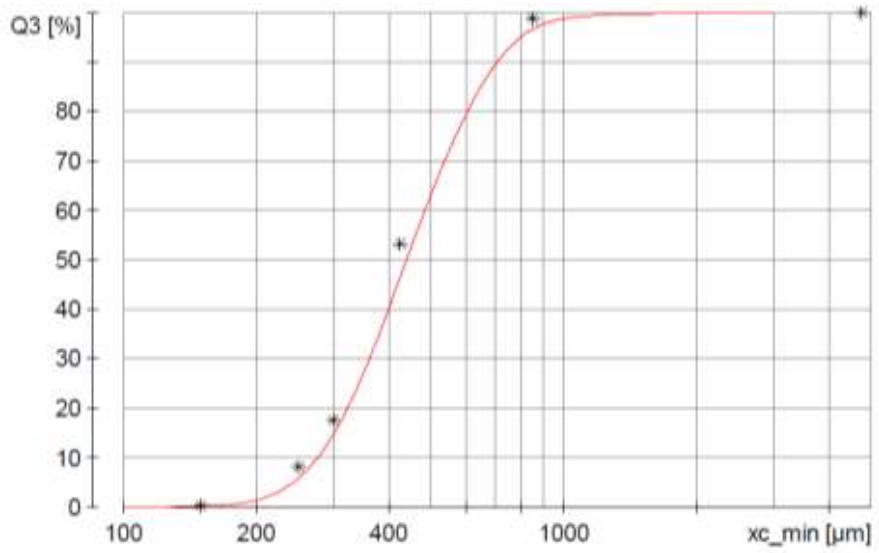
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Explosive Formulations by Particle Image Analysis

- Another sample was analyzed by Image Analysis and sieve sizes were selected based upon these results to better described the sample by sieving.
- Generally good agreement to sieving data
- Slight differences based upon orientation of particle when image acquired



RED – Cumulative Distribution (% passing)

* - Sieving Data

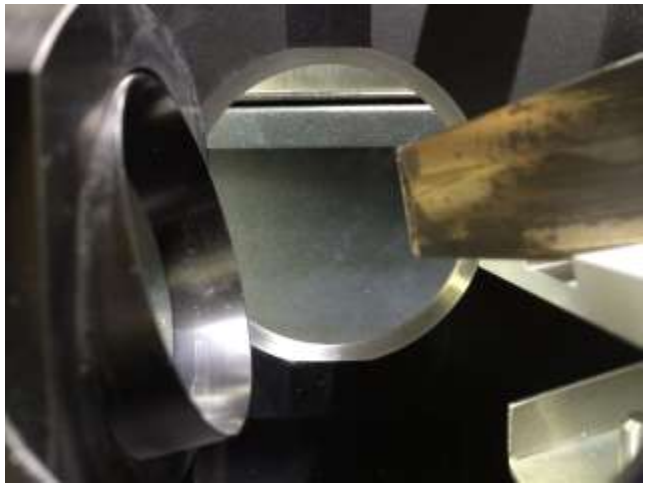
Sieve #	Sieving (% passing)	PIA (% passing)
#4	0.0	0.0
#20	1.2	3.3
#40	45.5	50.3
#50	35.7	31.7
#60	9.4	8.8
#100	7.9	5.7
PAN	0.3	0.2

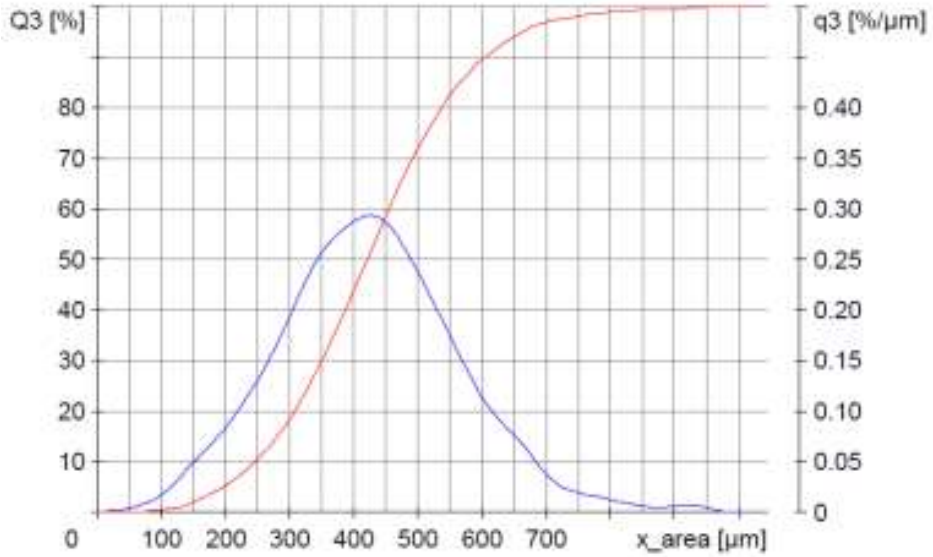
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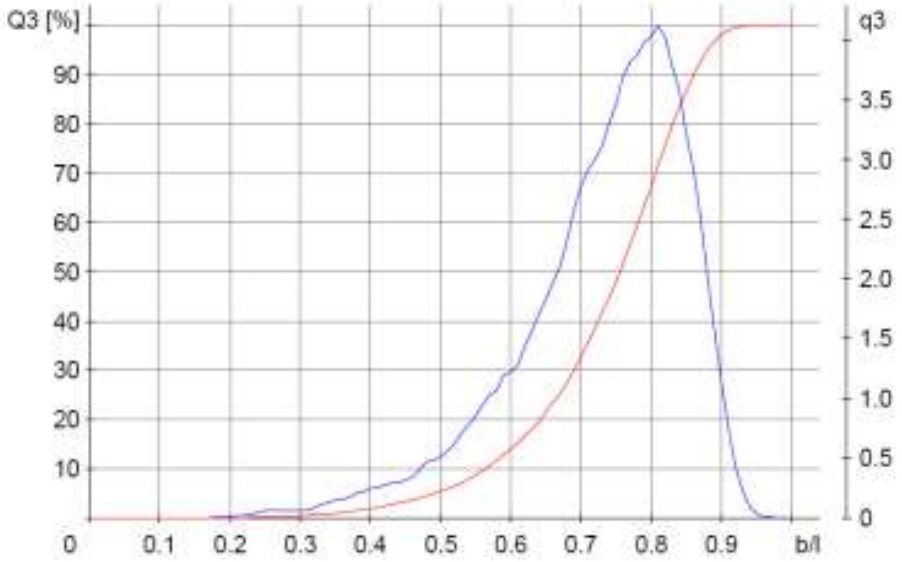
Energetic Materials by Particle Image Analysis

- Dry fine particles that agglomerate can be analyzed by vibration feed table and air pressure dispersion
- Particles are mixed with a stream of compressed gas to break up agglomerates.
- Particles and gas stream exit out of a nozzle past the camera for measurement.









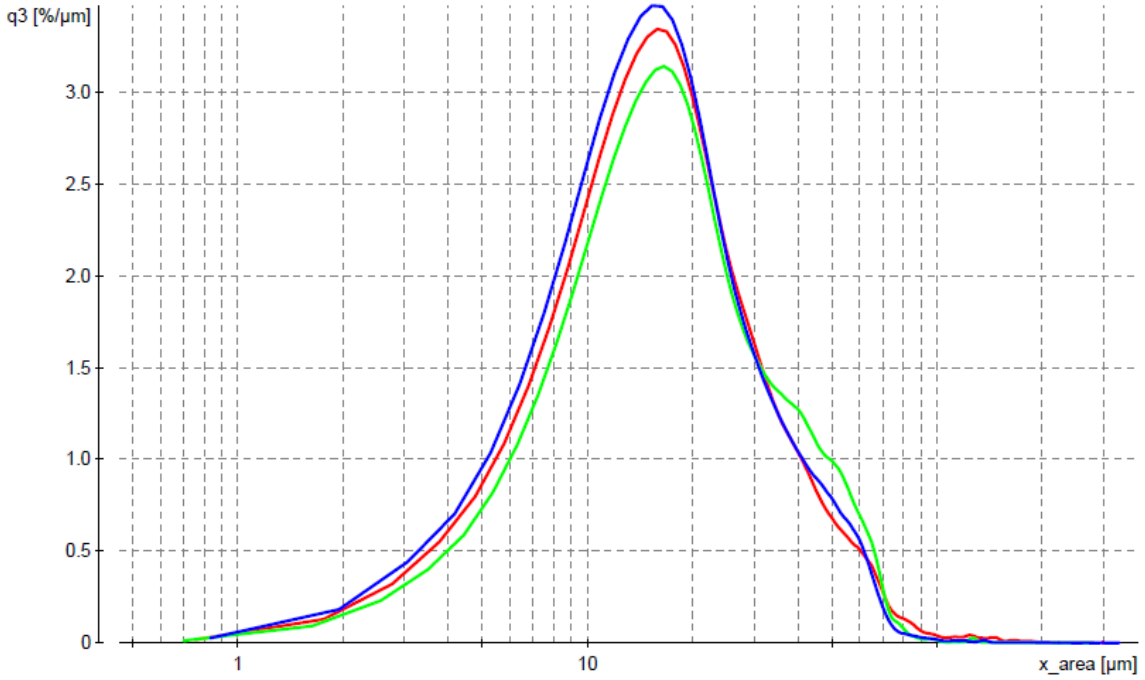
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Energetic Materials by Particle Image Analysis

- Fine particles that agglomerate can also be analyzed using a wet mode
- Particles are slurried in water. Agglomerates are broken up by sonication
- The slurry is pumped through a liquid cell past the camera for measurement.



Energetic Materials Methods Developed
TATB
NQ
NTO
RDX
HMX

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Summary

- Particle Imaging has been used to evaluate a variety of explosive formulations and ingredients.
- Imaging analysis has shown good agreement with sieving data for explosive formulations. Method is faster, less hands on, and easier to clean-up.
- Particle imaging analysis has shown the ability to distinguish differences in particle shape that other methods cannot detect.
- Data files can be stored and reprocessed to incorporate different discrete sizes in order to better characterize particle size for materials that have been shipped and for which a file sample is no longer available.

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AEROJET ROCKETDYNE

Advanced High-Performance Minimum Smoke Propellants

2021 Insensitive Munitions & Energetic Materials (IMEM) Technology Symposium



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Presenter: Cameron Lee

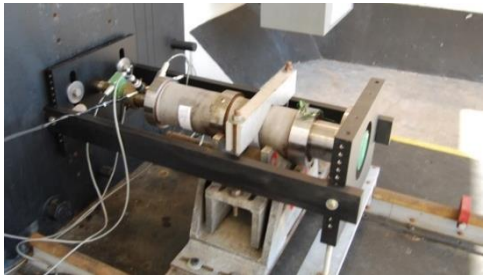
Project Objective and Impact

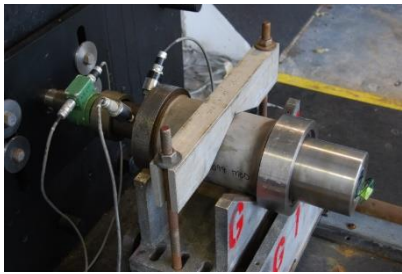
- The project objectives are to provide a minimum-smoke propellant formulation which will provide range improvement in a baseline system and provide Type IV fragment and Type V slow cook-off
  - The end-state of this project will provide the following:
    - Propellant + configuration giving range improvement
      - Increased I_{sp}, Increased I_{vol}, pressure, expansion ratio
    - Type IV fragment impact response
      - Energy partitioning (energetic binders)
    - Type V slow cook-off (at system level)
      - End-burning, Composite case + Forward and aft passive venting is critical for SCO
- Impact Summary
  - Current baseline minimum-smoke propellants exhibit Type I fragment impact response. Enhancements in fragment impact through lower sensitivity ingredients will provide IM enhancement without energy/performance degradation.
  - Target ingredient manufacture supported by continuous reactor technology for future manufacturing (MRL) improvement

Background – IHPRPT Phase III

- Under IHPRPT Phase III, high performance propellant development was conducted to meet NOL Class 1.3
- Ingredients/Formulation to JEMTP TRL-5
- Many ingredients screened for performance (aluminized system)
- Improved energetic materials found to add performance and reduce sensitivity - scaled to pilot plant level
- Shock sensitivity in NOL LSGT less than 70 cards
- Motor demonstration confirmed high efficiency and high specific impulse

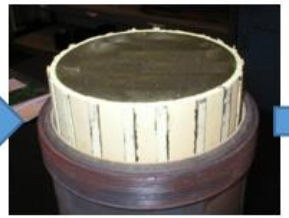






Technical Approach - Overview

- Technical Approach:
  - 1) Identify combinations of higher performance insensitive minimum-smoke ingredients based on heterocycles to meet performance,
  - 2) Characterize the downselected formulation and demonstrate useful mechanical properties, cook-off, and fragment impact characteristics at the subscale level, and
  - 3) Demonstrate performance in an end-burning heavywall demonstrator motor with features for desirable ballistic properties to match fielded system.

Insensitive Ingredients	Formulations	Motor Demonstrator	Motor Test
Year 1	Year 2	Year 3	Year 3
			

Goal of approach is to bring improved performance to the motor level

Motor Demonstration

- End-burning grain configuration selected for motor demonstration
  - Developed formulation from this effort incorporated into motor for higher performance
  - Finalized design to be addressed in year three; end-burning configuration being evaluated
  - Slow cook-off and fragment impact benefits from end burning grain configuration
- Two motor demonstrators to be considered:
  - HW 6” diameter (shown) preferred
  - HW 2.75” diameter uses less propellant (lowers need for experimental materials)





Motor demonstration planned in Year 3

Applications and Transition Potential

- The technology will provide increased performance with IM benefits through the use of demonstrated insensitive propellant ingredients. This will increase the range of a future tactical minimum smoke motor: Hellfire, TOW, or Javelin or similar variant missile system."
- This effort also complements other efforts: the insensitive propellant ingredients were initially used in aluminized systems with demonstrated performance increases.
- Other technologies, especially composite case, and energetic bonded closures could be combined with this technology.
- Target weapon system or systems are minimum smoke rocket motors such as Hellfire, TOW, and/or Javelin.

Questions?

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MSIAC
Munitions Safety Information Analysis Center

Supporting Munitions Safety



A Review of Mitigation Techniques for Small Calibre Munitions and Flares

IMEMTS 2021

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Aurihona Wolff
ENSTA Bretagne, Brest France



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Introduction

- In the IM world, mitigation techniques are techniques applied to the munition or its close environment to **reduce the probability** of a hazardous event occurring and to **reduce its impact** if it occurs.

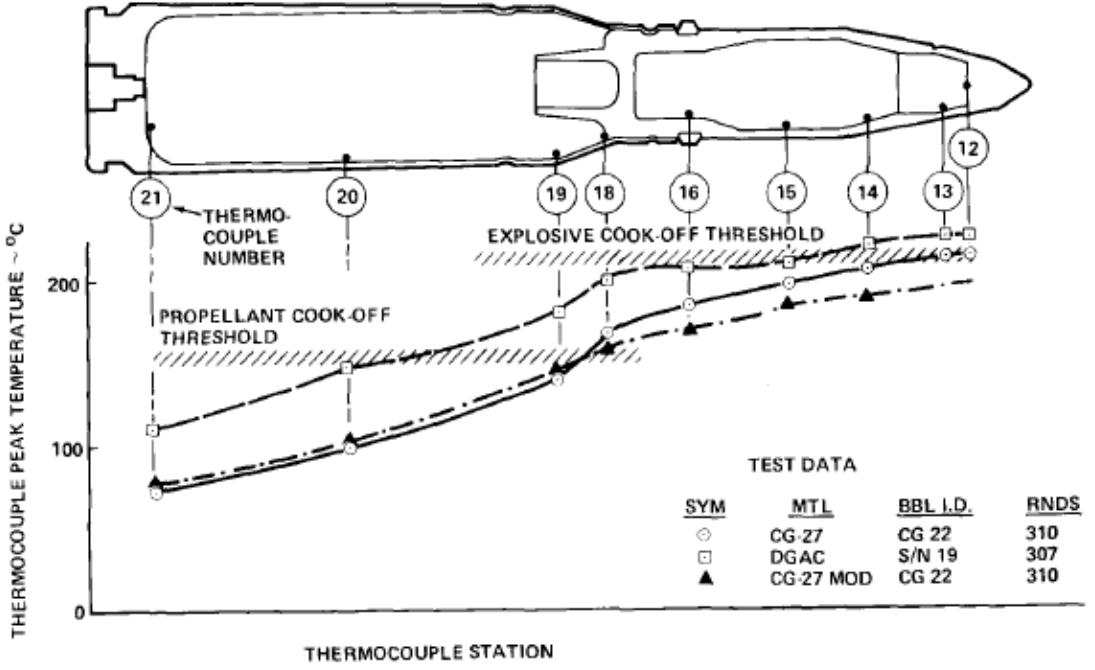
Energetic Material	System Design	Packaging
•New Crystals/Molecules •Crystal Morphology & Quality •Nano-Technology Materials •Advanced Binders •Energy Partitioning •Crystal Coating •Suppression Agent •Modeling & Simulation	•Liner Materials •Passive/Active Venting •Coating •Scoring •Thermal Protection Material •Barrier/Ballistics Material •Fuse & Initiators •Modelling and Simulation	•Passive/Active Venting •Storage Configuration •Packing Container Material •Thermal Protection Material •Barrier/Ballistics Material •Modelling & Simulation

- Contrary to mitigation techniques applied to warheads or rocket motors, mitigation techniques for **small calibre munitions** (20 mm and less, also called Small Arms Ammunition or SAA) and **flares** have suffered of a lack of attention.
- The review conducted at MSIAC in 2019-2020 aimed to fill this gap.

I. Mitigation Techniques for Small Calibre Munitions

II. Small Calibre Munitions: Threats

- In addition to the **IM threats** faced by other munitions (thermal threats, accidental reaction of nearby munitions, enemy aggressions), small calibre munitions may be subject to **bullet cook-off.**



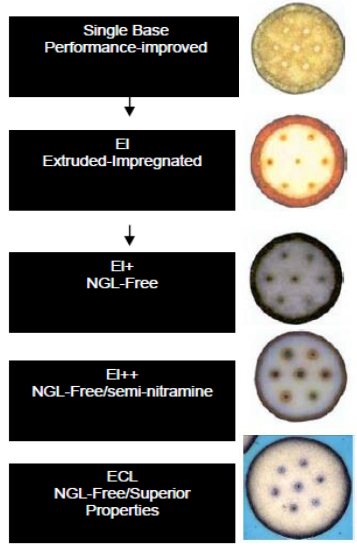
[1]

II. Small Calibre Munitions: Composition

- Use of a low vulnerability gun propellant composition (LOVA propellant) to decrease the vulnerability to **IM threats** and to **bullet cook-off.**
- Two main trends exist for less insensitive propellant formulations:

1. Nitramine-filled polymer-bonded compositions
   - Examples in this family include **Rowanite 317/318** (with a TPE binder, developed at BAE Systems, UK), **CLP-26 and CLP-15** (IMI, Israel), **NL100 and the NL0XX family** (EURENCO Bofors, Sweden).
   - An adaptation of the formulation/process would be required for small calibre applications

2. Modified Conventional NC-based compositions
   - NitroChemie (CHE) developed the extruded/impregnated propellant family (**EI®, EI®++ and ECL®**) with **less sensitive** but more expensive **energetic plasticizers** such as NENA. EI+ is said to present a **better cook-off behaviour** due to two layer surface coating.



[2]

II. Small Calibre Munitions: Composition

Additives can be used in the composition to **lower the flame temperature** and thus **decrease the thermal hazard**.

DHED	dihydrazine ethylenedinitramine
DANPE	1,5-diazido-2-nitraza-pentane
DADNH	1,6-diazido-2,5-dinitrazahexane
HZBTA	hydrazinium bitetrazolamine
MENENA	N-methyl-betanitroxyethynitramine
CG	cyanoguanidine
TAZ	triaminoguanidinium azide
UREA	urea

But, these additives may significantly **reduce performance and shorten the shelf life** of the munition by affecting its mechanical integrity.

II. Small Calibre Munitions: System Design

Venting Systems

- Examples exist on munitions of calibre down to 25 mm.
- A US Army effort to introduce venting holes in the design of 25 mm M910 cartridges resulted in an **improved IM response at fast heating and slow heating** over the baseline 25mm cartridges.

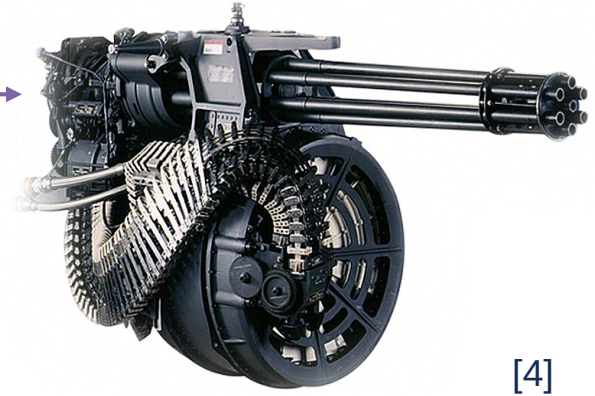


[3]

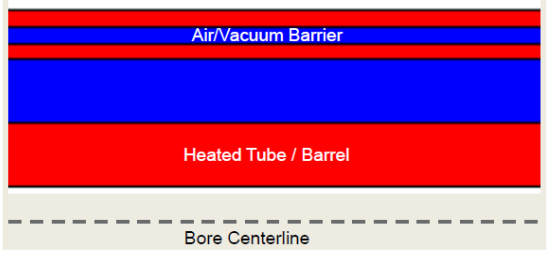
II. Small Calibre Munitions: System Design

Cooling Systems to Prevent Bullet Cook-Off

- Preventing bullet cook-off
  - Multiple barrels
    - Rotating barrels
    - Interchangeable barrels
  - Cooling agent
    - Internal cooling
    - Integral mid-wall cooling
    - External cooling
  - Distancing mechanisms
    - Moving away the rounds

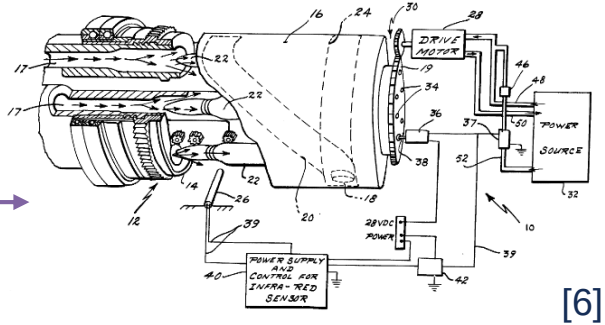


[4]





[5]

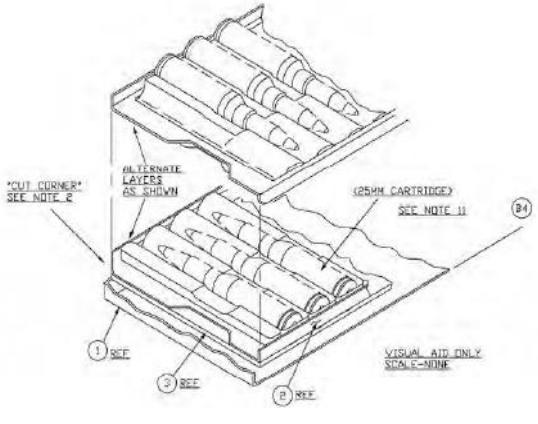


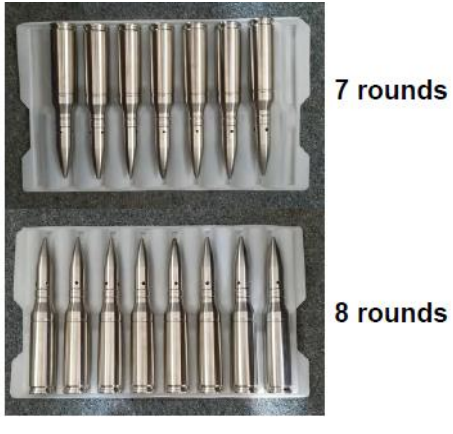
[6]

II. Small Calibre Munitions: Containers and Packaging

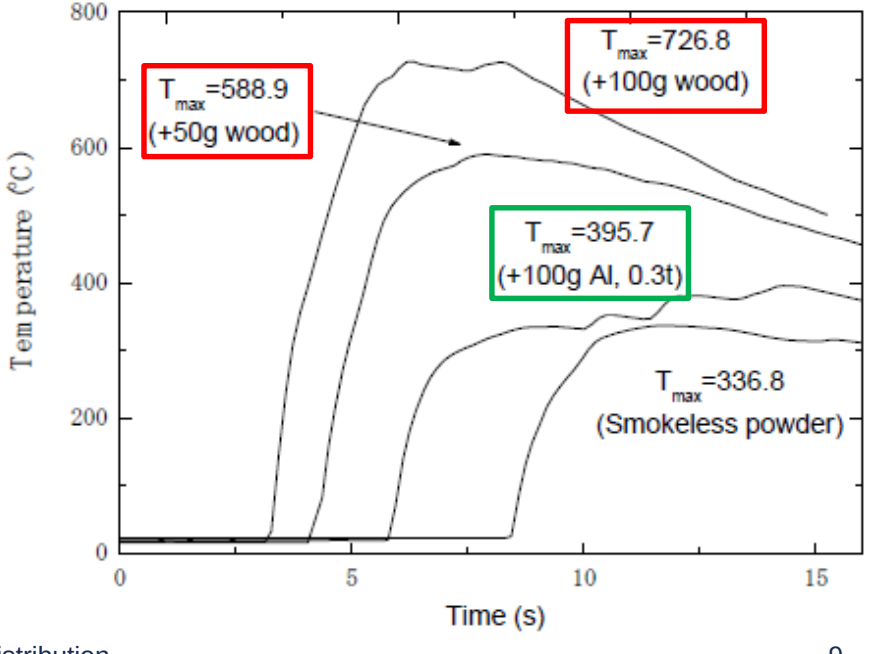
- Small calibre munitions can be transported in magazines or containers.
- A limited number of studies have been conducted on the **effect of the packaging** on the response of small arms ammunition to IM threats.

Multiple and delayed reaction at BI and FI due to HDPE trays in CNU-405/E containers [7]





Effect of the container material [8]



II. Small Calibre Munitions: Conclusion

- **Composition**: The sensitivity of small caliber munitions to IM threats and bullet cook-off can be improved by using **less sensitive formulations** and **additives**.

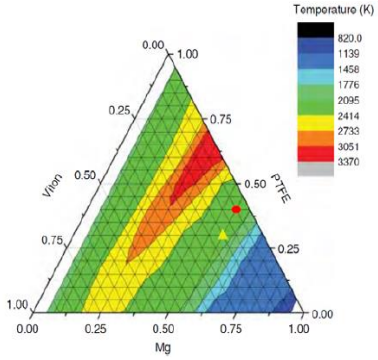
- **System Design**: **Existing technologies** that have been proved efficient for other types of munitions may also apply for SAA (e.g. venting, barriers or deluge systems) and could be implemented at a minimum cost.

- **Packaging**: The **container material** plays a major role in the reaction behaviour of the SAA. More studies are needed to better understand the contribution of the packaging on the munitions response.

III. Mitigation Techniques for Flares

III. Flares: Composition

- Traditional **MTV flares** are based on a Magnesium/Teflon/Viton with respective contents adapted to match the specifications
  - They are very sensitive to electrostatic discharge (ESD) and relatively sensitive to low energy impacts. The use of a specific binder, of pre-coated Alex®, or coating MTV with additives may reduce the sensitivity to ESD.
  - MTV flares are sensitive to bullet impact and sympathetic reaction threats.



[9]

- **Spectral flares** could be filled with Composite propellants, Double base (DB) propellants or Red-phosphorus (RP) based compositions
  - Safety and health hazards reported for RP based compositions in presence of moisture
  - Composite and DB propellants are much more hazardous than MTV flares

III. Flares: System Design

- As was proven efficient for other munitions, both **fire retardant painting** and **impregnation treatment of wooden boxes** can be used to delay the rate at which fire propagates through a stack of flares.

- Special liners can also be used to reduce a flare's sensitivity to fast heating threats, although they may affect the pyrotechnic performance of the flares.

- Kematal (a thermoplastic) or paper are found to be the most satisfactory materials for liners.

- More studies should be conducted on the thickness of the liner (minimum thickness?).

III. Flares: Packaging

- Attention must be paid to the material used for the **containers**:
  - Some experiments on pyrotechnics suggest that **wooden boxes** become severely damaged on drop trials and shocks and are consumed during fuel fire and bullet impact tests.
  - On the other hand, **metal containers** are likely to enhance explosive response to bullet impacts.
  - **Plywood packaging** around the outside of normal packaging could significantly increase the time to cook-off.

- **Dispensers**: Take into account the arrangement in a mixed magazine… or take advantage of it!





[10]

III. Flares: IM Test results

Review of IM tests and drop test results on MTV and spectral flares [11]:

IM Test/ Composition	MTV (CCM 218 Mk. 2)	MTV (MJU-10/B)	MTV (DSTL 22)***	MTV (undisclosed)	MTV (61.1:33.0:5)	MTV (unknown)	MTV (undisclosed)	MTV (undisclosed)	Spectral (DSTL 81)	Spectral (CCM 118 Mk.3 Type 3)	Spectral (DSTL 83)
Drop Test (Packaged)	No response @ 12 m		Safe @ 12 m						Safe for disposal @ 12 m		Safe for disposal @ 12 m
Fast Cook Off			IV		V at 527°C**			Non-detonation >430°C	IV		IV
Slow Cook Off			IV		No reaction @ 530°C. Pellets swollen by 1.2 mm**	Reaction at 700°C. Hardware not significantly damaged**		Deflagration /partial detonation >430°C		IV	
Bullet Impact (0.5" AP) (Packaged)	III @ 840 $m.s^{-1}$* IV/V @ 459 $m.s^{-1}$ III/IV @ 804 $m.s^{-1}$*	IV @ 567, 590, 721 & 808 $m.s^{-1}$	IV						IV (details not given)		IV (details not given)
Bullet Impact (7.62) (In dispenser)				IV @ 778 $m.s^{-1}$			IV or V @ 850 $m.s^{-1}$ Flares initiate with no dispenser damage				
Bullet Impact (8.58) (In dispenser)				IV @ 820 $m.s^{-1}$							
Bullet Impact (12.7) (In dispenser)							IV or V @ 900 $m.s^{-1}$				
Bullet Impact (14.5 API) (In dispenser)							IV or V @ 900 $m.s^{-1}$				
Sympathetic Reaction	No propagation (box to box)	No propagation (box to box)	No propagation (box to box)						No propagation		No propagation

*Blast overpressure was not measured in this trial, nor was an accurate measurement of bullet velocity.
**Testing conducted in Super Small-scale Cook-off Bomb (SSCB) and RARDE Small-scale Booster Cook-off Test (SBCT)
***DSTL 22 results are based on read-across from other countermeasure(s) with identical composition.

Warning
No comparison possible!

Note: No results available for RP based flares

II. Flares: Conclusion

- **Composition**: spectral flares have been proved to be much more hazardous than MTV flares due to the use of propellants designed for other applications. **Less sensitive alternatives** exist for composite and DB propellants but they may require adaptation studies to match the specifications of spectral flares.

- **System Design**: **Existing technologies** that have been proved efficient for other types of munitions may also apply for flares (e.g. fire retardants) and could be implemented at a minimum cost.

- **Packaging**: **Changing the packaging** is believed to be the easiest way of achieving a cheap and efficient IM solution for flares in the short term.

IV. The MTM database

The MTM Database: 391 examples, 443 references

ID	Description	Threats and Hazards	Components	Categories	Techniques	Permanent modification	Configurations	Ref.
2	Intumescent coatings to replace the NASA formulation EX-1C-82.	FH	Rocket Motor, Warhead	Bomb, Missile	Thermal protection	Yes	Packaging	5
436	Chrome or refractory metal plated bore	Cook-Off	Propelling Charge	Small Calibre	Thermal protection	Yes	System Design	498
437	Changing the MTV ratio in flare compositions	SH	Propelling Charge	Flare	Composition	Yes	Energetic Material	515, 519
438	Coating MTV	ESD	Propelling Charge	Flare	Composition	Yes	Energetic Material	516, 517

Shape Memory Polymer (SMP) to Disengage the End

Use of a piece of SMP to disengage the end of the rocket motor allowing therby the venting of the motor. It uses the same principle as shape memory alloy but using polymer instead of metallic alloy.



Description

References

Pictures

The MTMdatabase is accessible on the MSIAC secure website: https://portal.msiac.nato.int/

General Conclusion

- This review demonstrated that both **SAA and flares** were **under represented** in IM studies and corresponding examples of mitigation technologies. These are largely focused on high explosives and rocket propellants rather than on SAA and pyrotechnics.

- The reasons may be:
  - SAA and flares are small items that are difficult (if not impossible) to test individually → IM assessment of the container?
  - SAA and pyrotechnics, including flares, need to remain cheap items and mitigation techniques will need to be very inexpensive → Reuse and adapt existing technologies?
  - Regulations exist in some nations to exempt SAA and flares from IM assessment → Does it cover all situations? See next slide…

- Further information can be found in the MSIAC limited report L-249 and in the online database on mitigation techniques for munitions (MTM)

General Conclusion

Key figures for a convoy transporting troops and munitions (with SAA being by far the most represented category, in number) in Mali:



[12]

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U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

NATO Allied Ordnance Publication (AOP-7)

Policy, Data Requirements, and Tests for the Qualification of Energetic Materials for Military Use

Ken Tomasello, Bryan Baudler, Philip Samuels, Lori Nock

NDIA 2021 Insensitive Munitions and

Energetic Materials Technology Symposium

Distribution A

AOP-7 BACKGROUND

AOP-7 has been used for 30+ years to support energetic material Qualification and munition interoperability across NATO

- Ensure energetic materials (explosives, propellants, and pyrotechnics) meet fundamental safety requirements

Establishes Qualification policy and a standardized assessment approach

- Identifies various stimuli and test procedure requirements for all energetic types

- AOP-7 is used throughout NATO and non-NATO countries for acquisition (contract SOWs) and by national labs performing energetics Qualification

In the U.S., AOP-7 provides the basis for the establishment of DOD and service specific Qualification policy and direction

AOP-7 has not been updated and approved since 2003

QUALIFICATION POLICY SUMMARY

Energetic material Qualification approval is required as part of the process to certify munitions are safe for service use

- Required by numerous safety boards as part of independent approval process for munition certification

Two-phase process

- Initial Qualification phase assesses energetic material characteristics independent of an application
- Final (Type) Qualification phase evaluates energetics behavior in a specific system

Qualification approval or rejection is generally based on an evaluation of all test results in their entirety as opposed to the result of any one test

Qualification approval or rejection in the U.S. is the responsibility of Service Qualification Authorities in either the AF, Army or Navy/Marine Corps

- Delegated from OSD

INITIAL QUALIFICATION PHASE

AOP-7 is primarily focused on the initial Qualification phase

Requirements vary based on the energetic material class

- Explosives (main charge, primary, booster)
- Propellants (gun, solid rocket, liquids)
- Pyrotechnics

Data used to support Qualification:

- Small scale sensitivity test results (impact, friction, ESD, shock, etc.)
- Thermal sensitivity and characterization
- Mechanical property behavior
- Hazard Classification
- Ageing characteristics
- Performance attributes
- Toxicity
- Documentation (specifications, technical reports, certifications)

Multiple test procedures are acceptable and identified for the various stimuli

AOP-7 REVISION APPROACH

Multinational approach with Finland, France, Germany, Netherlands, Norway, Sweden, Turkey, UK, and the U.S. (all services)

AOP-7 has been re-organized to include a main body and three Standards Related Documents (SRDs)

- AOP-7 Main Body; Includes policy, requirements, and tests
- SRD-AOP-7.1; List of Qualified Energetic Materials
- SRD-AOP-7.2; National Test Requirements and Procedures
  - Addendum to AOP-7 requirements unique for each country
- SRD-AOP-7.3; National Authorities responsible for Qualification

SRD Benefits

- NATO revision process for ratifying an AOP or STANAG is fairly tedious
- SRD approval process provides streamlined approach appropriate for documents that require frequent revisions

The current revision process includes updates to AOP-7 and SRD-AOP-7.2 Qualification policy, test requirements, and technical test procedures

MAJOR CHANGES IN AOP-7 (7 DEC 2020 VERSION)

Updated technical requirements

- Revised required sensitivity stimuli, data, and test requirements
- Added new test STANAGs used for Qualification testing
- Updated aging protocol, booster explosive, and liquid propellant Qualification

Added Recommended tests

- Tests and analyses not essential but should be considered for inclusion as part of Qualification
- Support in-depth energetic material characterization
- Provide data supporting ageing or weapon performance

Clarified requirements and definitions for:

- Handling and release of Qualification data, Intellectual Property Rights (IPR) and Non-Disclosure Agreement (NDA) Details
- Qualified energetic materials may need to be re-qualified where new processes, manufacturers, or ingredient sources are introduced
- Added requirement that nations may conduct periodic audits of in-service Qualified energetic materials – this could lead to Qualification cancellation

Updated standard NATO definitions and annexes that provide a recommended Qualification certificate, references, and AOP-7 history

AOP-7 TABLE 1 – GENERAL REQUIREMENTS FOR ENERGETIC MATERIAL QUALIFICATION

Requirement	Type of Information Required
1. Details of the composition and its ingredients per the material specification	Composition ingredients, ingredient percentages in formulation with allowable tolerances, particle size, final product and ingredient sources.
2. Intended role and energetic material type	Main charge high explosive, booster explosive, primary explosive, pyrotechnic composition, gun propellant, solid rocket propellant.
3. Physical form as used in-service and processing characteristics	Method of manufacture. Pressed, melt-cast, cast-cure, extruded, machinability characteristics, specialized handling considerations.
4. Chemical and physical properties	Theoretical maximum density, recommended density for applications, melting point (for melt-cast explosives), growth and exudation properties, chemical characterization and compatibility, hygroscopicity, vapour pressure (for liquids), etc.
5. Sensitivity, mechanical, and thermal characteristics	Sensitivity (impact, friction, ESD, shock), thermal properties (reaction/decomposition temperatures, vacuum stability), mechanical properties.
6. Performance characteristics	Detonation velocity, critical diameter, burning rate, etc.
7. Comparisons to other similar compositions (if any) already in service	Comparisons to energetic materials with a safe history of service use. Includes sensitivity, performance, and mechanical properties.
8. Information about any known incompatible materials	List of substances (e.g., polymers, plasticizer, lacquers, oxidizing agents, etc.) that should not be kept in contact with the energetic material.
9. Toxicity data, environmental safety, and compliance with National Health and Safety requirements	Material Hazard Data Sheet, Safety Data Sheet, Hazard Classification, toxicity information, environmental safety, etc.

AOP-7 TABLE 2 – SPECIFIC QUALIFICATION DATA REQUIREMENTS

Test / Assessment	Explosives			Propellants			Pyro-technics
	Primary	Booster	Main Charge	Gun	Solid Rocket	Liquid	
Sensitivity Testing							
- Impact Sensitivity	M	M	M	M	M	M	M
- Friction Sensitivity	M	M	M	M	M	M	M
- Electrostatic Sensitivity	M	M	M	M	M	M	M
- Shock Sensitivity	R	M	M	R	M	M	
- Adiabatic Compression						M	
Thermal Characterization							
- Thermal Analyses (DSC/DTA/HFC/TGA)	M	M	M	M	M	M	M
- Thermal Stability	M	M	M	M	M	R	M
- Chemical Compatibility	M	M	M	M	M	M	M
- Self-Heating Critical Temperature		M	M			M	
- Auto Ignition Temperature		M	M	M	M		
- Growth/Exudation		M	M				
- Thermal Sensitiveness and Explosiveness		M	M	M	M		M
- Heat Capacity/Thermal Conductivity		R	R	M	R		
- Flash Point/Vapor Phase Ignition						M	

M = Mandatory; R = Recommended

Note: This Table may change slightly in the SRD national sections.

AOP-7 TABLE 2 – SPECIFIC QUALIFICATION DATA REQUIREMENTS

Test/Assessment	Explosives			Propellants			Pyro-technics
	Primary	Booster	Main Charge	Gun	Solid Rocket	Liquid	
Chemical Characterization	M	M	M	M	M	M	M
Mechanical Property Characterization		M	M	R	M		
Performance Characterization							
- Detonation Velocity		M	M				
- Critical Diameter		M	M	R	M	M	
- Burning Rate				R	R		R
- Unreacted Hugoniot		R	R				
- Run-to-Detonation		R	R				
- Detonation Products Equation-of-State		R	R				
- Detonation Diffusing and Spreading		R	R				
Ageing Protocol, Plan, and Data	M	M	M	M	M	M	M
Hazard Classification	M	M	M	M	M	M	M
Toxicity	M	M	M	M	M	M	M

M = Mandatory; R = Recommended

STANAGS USED FOR QUALIFICATION

Many STANAGs applicable to Qualification testing were developed with the goal of replacing national testing standards

- Numerous test procedure STANAGs exist for sensitivity, thermal, and mechanical property characterization
- Goal was to identify a single test that all nations would perform
- Found to be impractical

Could not accommodate specific national procedures, apparatus, technical limitations, historical data sets, in performing the test

- Unable to make a strong case that any one specific test was superior to others

Multiple test methods for specific stimuli included as a compromise

- Approach is to calibrate test procedures using standard reference materials (ex. RDX)
- Results are also compared to Comparison Energetic Materials with a history of safe in-service use

Agreement within each STANAG is “participating countries……have decided to accept each test as a standard procedure for determining the XXXX sensitivity”

SRD-AOP-7.2 U.S. SECTION UPDATE (MARCH 2021)

Updated Service Qualification Authorities (SQA)

- Emphasized need to meet with SQA tech reps prior to starting a Qualification program
  - Obtain clarification and guidance on test plan and approach
  - Critical towards developing aging program and need to perform recommended tests
- Emphasized OSD policy emphasizing use of STANAGs/AOPs for acquisition

Updated Qualification technical requirements

Revised Test Procedures – minimize number of tests for each stimuli

- Standardized test procedure format
- Updated existing procedures to include additional test details, configuration, typical results, and references
- Added new procedures that reflect more modern and mature analytical techniques
- Eliminated tests that were obsolete or not relevant to address Qualification requirements
- Updated Comparison Energetic Materials used to compare results from different procedures for the same stimuli

Expansion of ageing protocol, mechanical property, and performance tests details

Updated and clarified booster explosive pass/fail criteria

SRD-AOP-7.2 TEST DESCRIPTION FORMAT

Standardized Format for Test Descriptions

- Type of Test: Describes stimuli being assessed (ex: impact sensitivity)
- Purpose: Top-level explanation of why the test is performed
- Test Background: Historical summary
- Test Arrangement: Detailed description of test apparatus and set-up. Schematics of test configuration provided where appropriate
- Procedures: Specific test procedure
- Results and Data: Common test result observations and reporting requirements
- Supplemental Information
- References

Goal is to develop test procedures that could be used at any facility as written without modification or need to rewrite document

- Provide as much detail as possible

Slight site changes may be required to conform with local requirements (safety, facility layout, personnel requirements, etc.)

TEST REQUIREMENTS SRD-AOP-7.2 (MARCH 2021)

Section 100.1 Impact Sensitivity Tests (4)

100.1.01	ERL Impact	100.1.02	Ball Drop Impact
100.1.03	BAM Impact	100.1.04	mBOM Impact
100.1.05	JANNAF Impact Test		

Section 100.2 Friction Sensitivity Tests (2)

200.1.01	ABL Sliding Friction	201.02.00	BAM (Julius Peters) Friction

Section 100.3 Electrostatic Sensitivity Tests (1)

100.3.01	ABL Electrostatic Discharge

Section 100.4 Shock Sensitivity Tests (5)

100.4.01	NOL Small Scale Gap Test	100.4.02	NOL Large Scale Gap Test
100.4.02	Expanded Large Scale Gap Test	100.4.03	Super Large Scale Gap Test
100.4.05	IHE Gap Test	100.4.06	Adiabatic Compression

TEST REQUIREMENTS SRD-AOP-7.2 (MARCH 2021)

Section 200: Thermal Characterization (15 Tests)

- Includes Differential Thermal Analysis (DTA), Differential scanning Calorimetry (DSC), Heat Flow Calorimetry (HFC), etc.

Section 300: Mechanical Property Characterization (6 Tests)

- Uniaxial Compressive Strength, Tensile Strength, Coefficient of Thermal Expansion, etc.

Section 400: Performance Characteristics (8 Tests)

- Detonation Velocity, Critical Diameter, Strand Burning, etc.

Section 500: Physical Properties, Hazard Classification, and Miscellaneous (5 Tests)

- Density, Specific Gravity, Cap Test, etc.

Section 600: Aging Assessment and Evaluation

- Multi-phase approach – still in development
- Will reference test procedures for sensitivity, thermal, and mechanical property characterization

REMAINING SRD TASKS

Finalization of Aging protocol requirements

Review of statistical processes to determine pass/fail criteria for booster explosives

- May be function of specific test procedures

Development of standardized test procedures for Recommended performance tests

Completion of several mechanical property test procedures in conjunction with NATO working group

Review and inclusion of potential changes to standardized shock sensitivity tests

WHAT'S NEXT?

Main body of AOP-7

- Issue next Draft in June 2021 based on Dec 2020 version
- Perform final walk-through at the Fall 2021 AC/326 SG/A meeting
- Incorporate changes in a Final for ratification as part of Spring 2022 AC/326 SG/A meeting

U.S. National Section (SRD-AOP-7.2)

- Adjudicate comments to March 2021 Draft and issue next version in July 2021
- Expect to need one more revision before developing a Final document later in 2021
- Depending on extent of input, goal is to complete the document by the end of CY21

Additional input from energetics community is welcome and highly encouraged

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Land Armaments Directorate



IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

- James Padfield (RWM Italia)
- Philip Samuels, Keyur Patel, Scott Faluotico, and Erik Boykin (US Army CCDC DEVCOM)
- Lt Col. Francesco Rutigliano and Capt. Fabio Formato (Land Armaments Directorate)

NDIA Insensitive Munitions & Energetic Materials Technology Symposium, April 2021

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IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Contents

- Background to the programme
- Modernisation of the DM111 design
  - Explosive materials
  - Other IM features
- Loading process
  - Upgrades to RWM Italia melt-cast filling plant
  - Development of the loading process
- Tests of loaded rounds:
  1. Reliability of the initiation train
  2. Fragmentation/arena trials
  3. IM tests

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IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Introduction

- The Land Armaments Directorate (*Direzione Armamenti Terresti, DAT*) are the department within the *Ministero della Difesa* in Italy responsible for technical development and procurement of artillery
- DAT asked RWM Italia to demonstrate the feasibility of reloading its stock of DM111 projectiles, which are currently loaded in Composition B
  - 64 × DM111 rounds were demilitarised and provided to RWM Italia
- In October 2015, RWM Italia signed a **Cooperative Research and Development Agreement** (CRADA) with the US Army Combat Capabilities Development Command (CCDC DEVCOM) at Picatinny Arsenal (formerly known as ARDEC)
- CCDC DEVCOM and BAE Systems OSI at Holston have developed a range of less sensitive melt-cast explosives:
  - Large-calibre artillery (105 and 155 mm): **IMX-101** to replace TNT fills
  - Mortar ammunition (60 and 81 mm): **IMX-104** to replace Composition B fills
  - Large improvement in IM characteristics, while maintaining lethality for the user

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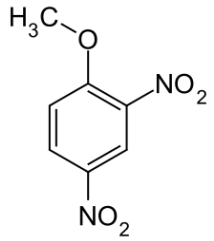
IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Modernisation of the DM111 Design: Explosive Materials

- Existing projectile utilises Composition B with an RDX/wax booster
- Energetic materials upgraded to less sensitive explosives
- **Main charge explosive in IMX-104**
  - Melt-cast: same infrastructure as TNT/Comp B filling

NTO	DNAN	RDX
53 %	32 %	15 %

- **Removable booster (Supplementary Charge) in PBXN-9**
  - 92 wt% HMX-based, pressed explosive composition
  - Successful history in munitions that perform well in IM tests
    - Including in the US Army's 155 mm artillery rounds
  - Booster can be removed to allow use of a deep intrusion fuze



2,4-dinitroanisole (DNAN)



IMX-104 flakes



PBXN-9 booster

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Modernisation of the DM111 Design: Lifting Plug

- Use of meltable ionomeric plastic fuze plug
  - Melting point ~125 °C
  - Provides a venting path in cook-off scenarios (as we shall see later)



Old style steel plug



Meltable plastic plug



Ejected plastic plug after FCO test

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Development of the IMX-104 Melt-Cast Loading Process

- Upgrade of RWM Italia's melt-cast filling plant at Domusnovas in Sardinia
  - Higher m.p. of DNAN-based explosive (95 °C) vs TNT-based explosive (80 °C)
- Filling tooling:
  - Water-filled carts and cooling chambers to cool in a controlled manner
  - Ensures high-quality casts (free of piping, shrinkage porosity, etc.)
- Development of process for radiographic examination process
  - To ensure that we are getting high quality X-ray images so that we're sure we're actually seeing any defects present in the cast







Result: rounds meet the applicable requirements for cast quality in 155 mm projectiles

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Testing Phase 1: Reliability of Initiation Train

- Essential to show that:
  - The fuzes already in service with Italian Army reliability initiate new PBXN-9 booster
  - And also that the PBXN-9 booster initiates the IMX-104 main charge
- Series of tests carried out at the DAT artillery test centre at Nettuno near Rome, across the range of anticipated operational temperatures:
  - Cold (-46 °C); ambient temperature; hot (+63 °C)
- Instrumentation (blast overpressure and high speed video) provided by DAT
- **All tests showed full detonation of the explosive train**





0.0 ms 0.1 ms 0.2 ms 0.3 ms

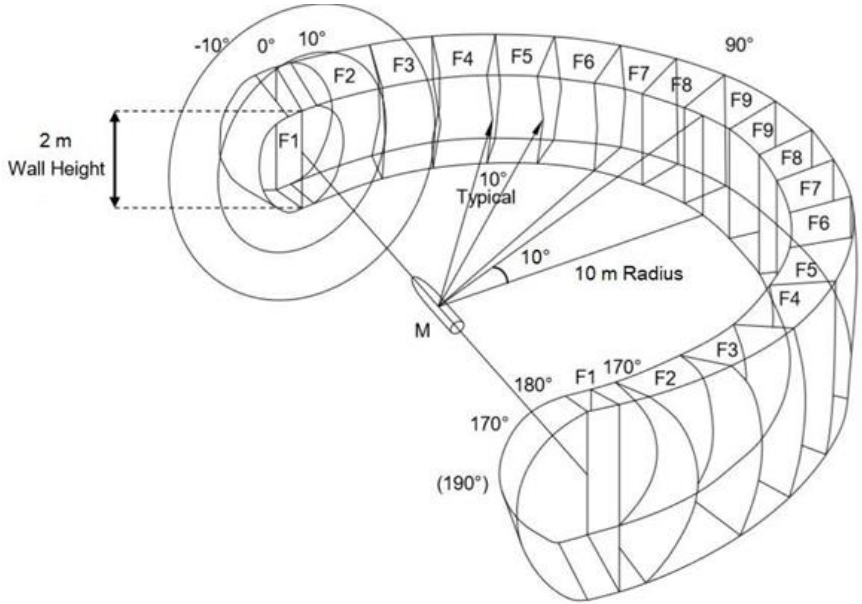
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Testing Phase 2: Fragmentation/Performance Tests

- Full fragmentation arena trials, as per ITOP 4-2-813, were carried out at COTEC





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IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Testing Phase 2: Fragmentation/Performance Tests

- Full fragmentation test results are **CLASSIFIED**
- But we can say:
  - Fragment velocities slightly lower than L15/Comp B, but this was expected
  - Spatial and mass distribution of fragments also slightly different
- But **overall**, lethality of the DM111/IMX-104 is equivalent to that of the old projectile

	Composition B	IMX-104
Velocity of Detonation	7900 m/s	7400 m/s
Pressure of Detonation	260 kbar	240 kbar

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Testing Phase 3: IM Tests: Fast Cook Off

- Liquid fuel fire test in accordance with AOP-4240 at COTEC (UK)
- Plastic lifting plug and booster ejected and recovered a few metres from the hearth
  - No reaction of the PBXN-9 explosive in the booster
- IMX-104 main charge burned out in around 10 minutes









Type V - Burning

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Testing Phase 3: IM Tests: Slow Cook Off

- Test carried out at 15 °C per hour as required by AOP-4382 at COTEC (UK)
- First signs smoke seen at approximately 190 °C (oven temperature)
- Explosive burned from the top down in around 35 minutes
  - (Temperature plot appended to this presentation)
- Plastic lifting plug not ejected – merely softened and hot gases vented around it







Type V - Burning

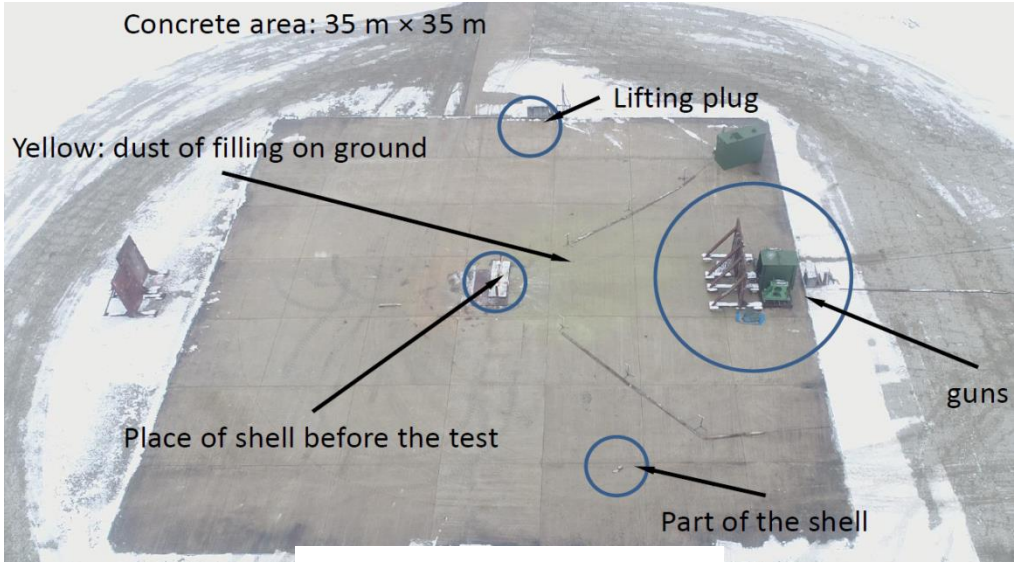
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Testing Phase 3: IM Tests: Bullet Impact



- Test performed at RWM Unterlüß (Germany)
- Three-round burst of .50" (12.7 mm) AP ammunition
- No evidence of reaction of the explosive
- But steel lifting plug (750 g) was ejected ~19 m
- Possibly Type V reaction with plastic lifting plug





Type IV - Deflagration

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IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Testing: Summary

1. Initiation tests show that the new explosive train functions with the Italian Army's in-service fuzes across the range of operating temperatures
2. Lethality is equivalent to the existing in-service L15/DM111 rounds loaded in Composition B
3. But the DM111 projectile loaded in IMX-104 performs significantly better in the Insensitive Munitions tests

	Fast Heating	Slow Heating	Bullet Impact
Requirement from STANAG 4439	*Type V*	*Type V*	*Type V*
DM111/IMX-104	**Type V**	**Type V**	**Type IV**
L15/Comp B (from MSIAC AIMS database)	Type III	Type II	Type III*

* BI Test result for an M107 projectile loaded in Composition B, rather than an L15

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IM Upgrade of Italian Army DM111 155 mm Artillery Projectile

Acknowledgements

- Italian Ministry of Defence:
  - Donation of the old DM111/Comp B rounds
  - Use of the DAT firing range, instrumentation, and facilities at Nettuno
- The US Army CCDC DEVCOM technical team:
  - Scott Faluotico and Erik Boykin for development of the IMX-104 loading process
  - Scott McClain for development of the X-ray process
  - Phil Samuels and Keyur Patel for advice on the selection of explosive materials, and on the test procedures and methods
  - Lethality Analysis Group for advice with the analysis of the fragmentation tests
- RWM Italia:
  - Fabio Mantega and his Production Line B team at Domusnovas, who actually did all the hard dirty work of loading the rounds, machining the fuze cavities, etc.

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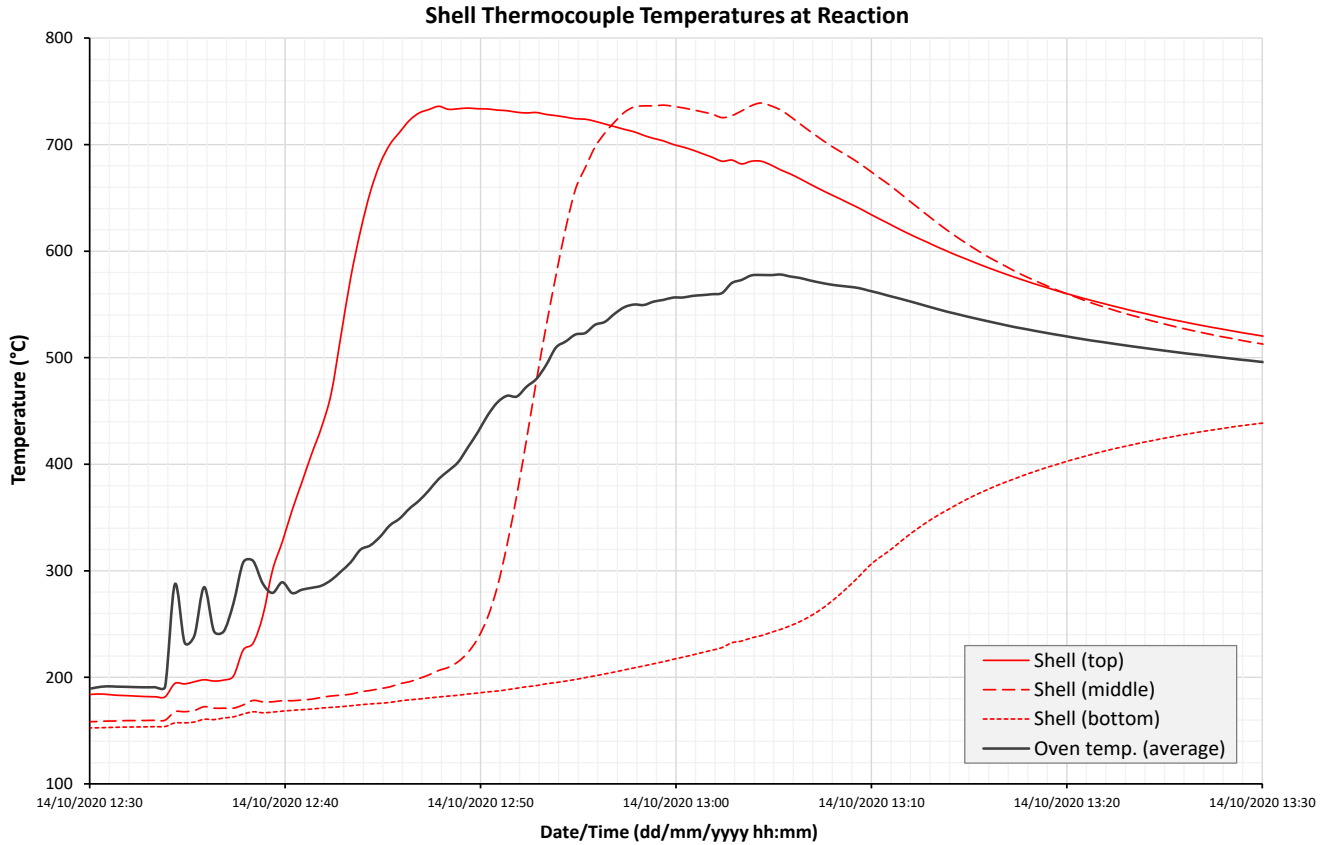


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IM Tests: Slow Cook Off temperature plot



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NAVSEA
WARFARE CENTERS
DAHLGREN

Gun and Electric Weapon Systems (E)

Barrel Heating for Hot Gun Cook-Off Thermal Analysis

Paper Number 23501

2021 Insensitive Munitions and Energetic Materials Technology Symposium

Jon Yagla and George Anderson

7-8 April 2021

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Hot Gun Safety

A fire mission may call for hundreds of rounds of ammunition to be expended in a short period of time. The "hot gun" safety concern is for a round to be loaded into the gun, and fail to fire for some reason. If the round cannot be ejected it may cook off in the chamber. The picture on the left shows an explosion in a 16-inch gun turret. The left gun of the 5"/38 cal. mount (right) was thrown 900 ft. into the sea when a projectile detonated in bore. Guns are also installed in aircraft and fighting vehicles, where the there is no place to escape or hide.



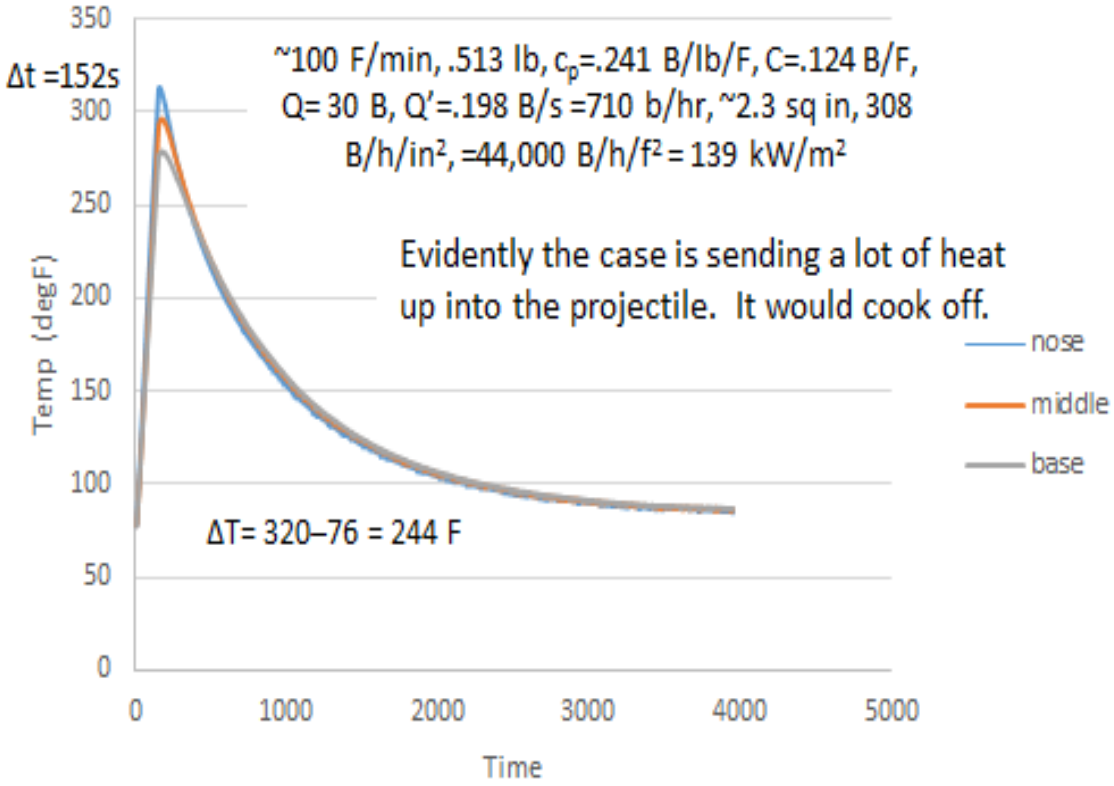


Laboratory Experiment With a Cartridge in a Radiant Chamber



Cartridge With Thermocouples in Radiant Chamber

Sample calculation of the heat flux into a round in the radiant chamber from measured temperature



The heat flux of 139 kW/m^2 is what we see in fast cook-off testing

Stages of Heating and Modeling

- Action time – projectile starts from rest and travels down to the muzzle exit plane
- Blowdown – hot propellant gas vents out of the barrel causing additional heating
- Heat Soaking – barrel accumulates heat which decays only slightly between rounds

Heating starts when the projectile first begins to move. Pressure forces the projectile into the bore. For small arms the bullet jacket is "engraved" at this time. For larger calibers, driving bands of metal or plastic are used. The engraving process happens quickly, and there is plastic deformation of the band that causes heat. Since the band is in contact with the barrel, heat flows into the barrel at this time. It also flows into the projectile body. Pressure on the base of the projectile and hydrostatic pressure caused by acceleration cause the projectile to swell and push the barrel radially outward, creating frictional heating of the barrel and the projectile. The torque applied to the barrel causes frictional heating as the band slides along the shoulders of the rifling lands. It takes great force to push a fired projectile back through the barrel, indicating large friction is present.

Internal ballistics models are used to move the projectile down the barrel during the action time. The velocity, viscosity, flame temperature, bulk temperature, and pressure are computed as functions time and distance. From the Reynolds numbers and Nusselt numbers, convection to the barrel can be calculated. Radiation is calculated from the Stephan-Boltzman equation.

Blowdown starts when the projectile band crosses the muzzle exit plane. The velocity, temperature, and pressure in the barrel can be calculated using formulas developed for designing muzzle brakes. The barrel vents over a time period much longer than the action time. Even though the heat fluxes are smaller, they act over a longer period of time and provide a comparable amount of heat.

Outline

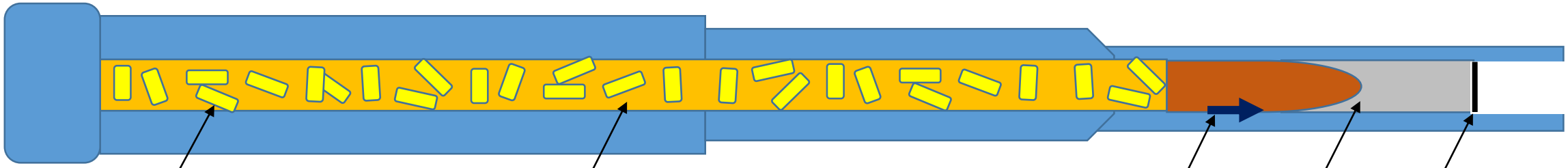
Action time – In bore period. Projectile starts from rest and travels down to the muzzle exit plane
5 slides

Blowdown – Hot propellant gas vents out of the barrel Convection and Radiation
3 slides

Lumped Parameter Models – complete math model of a barrel with a projectile
3 slides

Current Experiments – Instrumented 105mm barrel and projectile
2 slides

Sources of Heat in Barrel



Propellant grains burning at flame temperature T_f

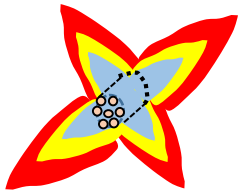
Propellant gas at "bulk" temperature T_b

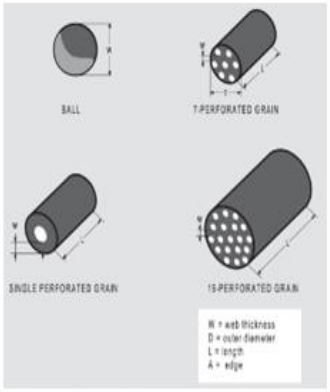
Friction

Compressed air at Temperature T_s

Shock wave

Luminous Particles and Chemistry



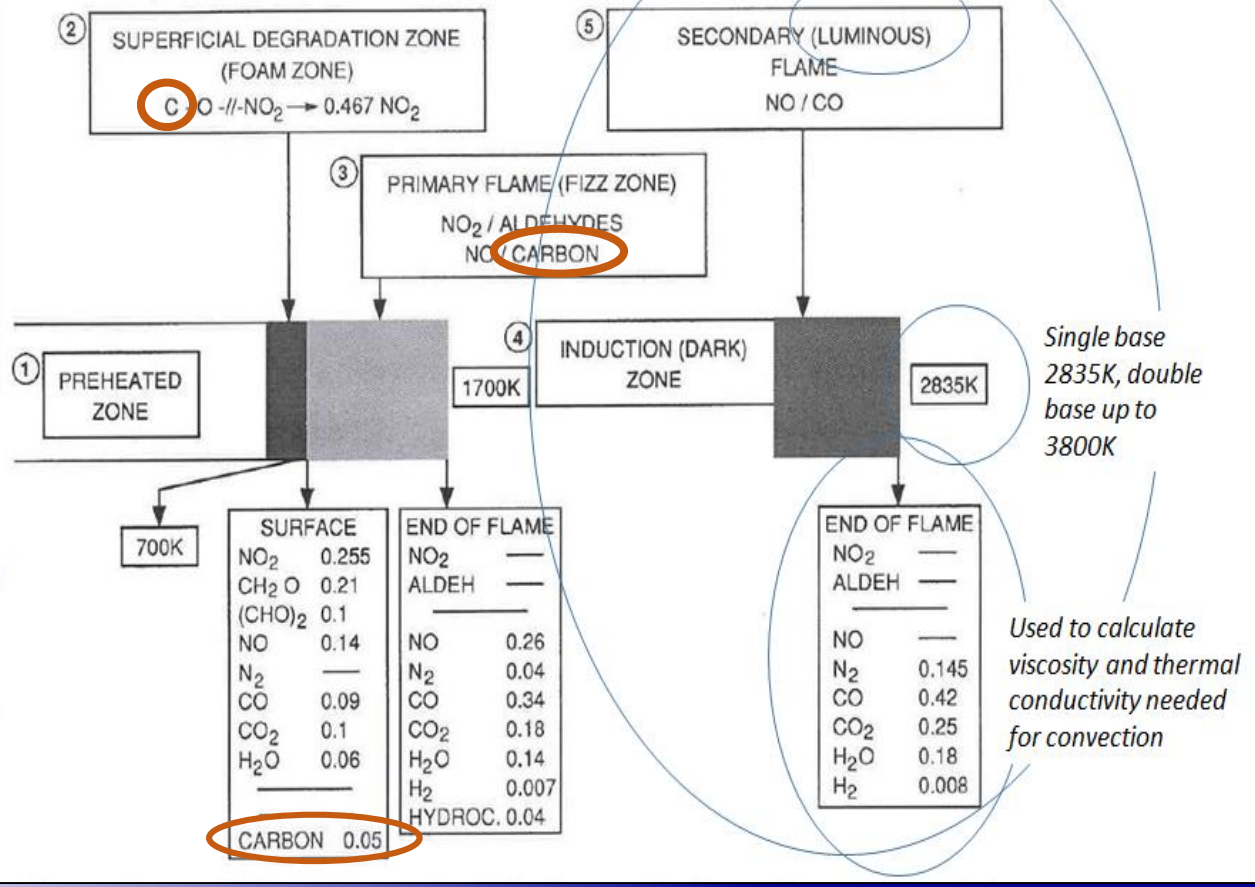


Propellants are designed to burn at a nearly constant rate. As the outer surface area diminishes, the inner area increases. The radiation output should be nearly constant. (jjy)

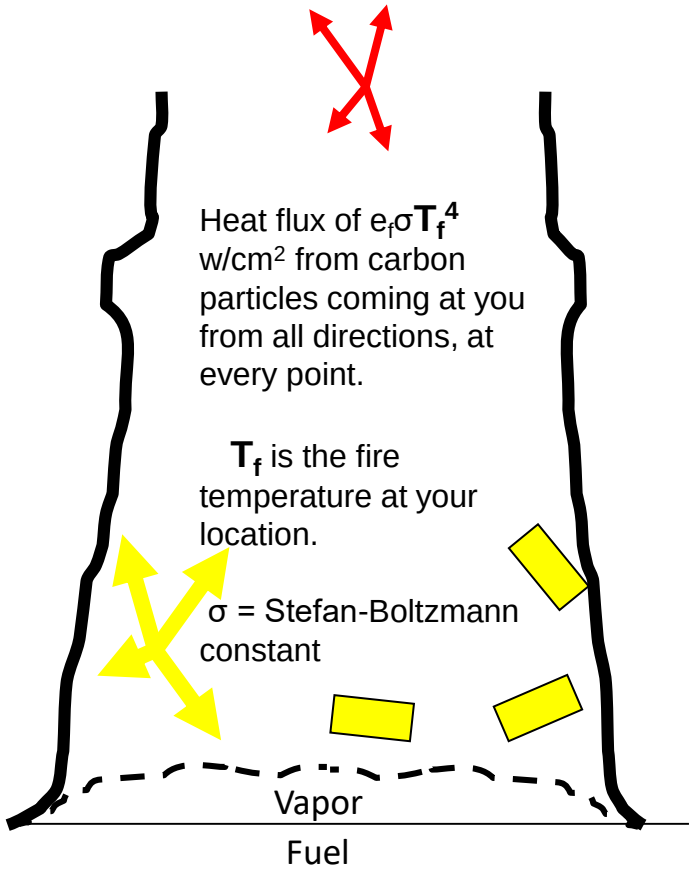
https://www.vihtavuori.com/tech-blog-powder-grain-shapes/

Combustion of Solid Propellants

G. Lengellé, J. Duterque, J.F. Trubert
Research Scientists, Energetics Department
Office national d'études et de recherches aérospatiales (ONERA)
29 avenue de la Division Leclerc
BP 72 – 92322 Châtillon Cedex
FRANCE



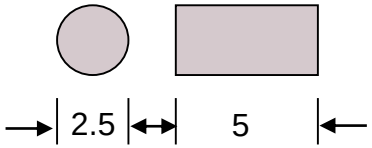
Fast Cook-Off : *90% of the heat is radiation, 10% is convection*



The same formulas are used for gun barrel heating

Worked Example – Heat absorbed by a cylinder in a 1750 deg F Sooty Fire

$e_f\sigma T_f^4$ B/hr/ft^2 emitted from fire in all directions

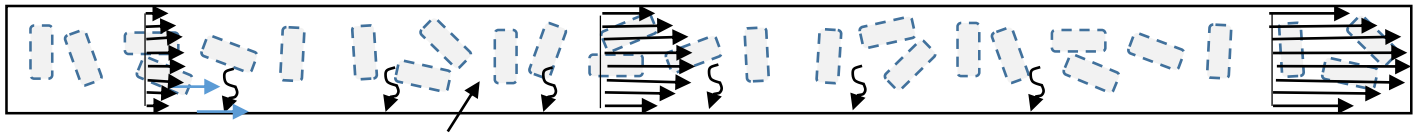


e_b= 0.9

Stefan-Boltzman Constant	σ	1.7140E-09	B/hr/ft^2/deg R^4
Emissivity of fire	e_f	1.00	
Temperature of fire	T_f	1750 +460 =2210 R	
Emitted radiation	**q”**	1.1144E+04	B/hr/ft^2
Surface area of Cyl.	**A**	.273 ft^2	
Total incident radiation on cylinder	**q’**	11,100 B /hr	
Absorbed for e_b=0.9		**10,000 B/hr**	

Gas Dynamics Mechanisms for Heat Transfer to Barrel During Action Time

Convection



Velocity ~ 0, no convection

T_{bulk}

Gas temperature

$q'' = h_c (T_{bulk} - T_{gun})$

h_c = convection coefficient

Velocity = Vp, maximum convection

Small, < 5% of total heating

Radiation from gas at T_{bulk}



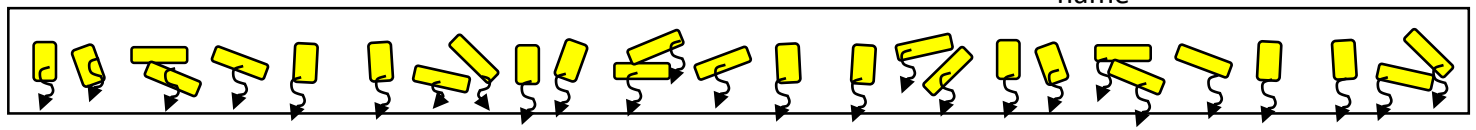
T_{bulk}

$\varepsilon\sigma T^4_{bulk}$

$T^4 \sim 2835^4$

About 30% of radiation heating

Radiation from propellant grains at T_{flame}

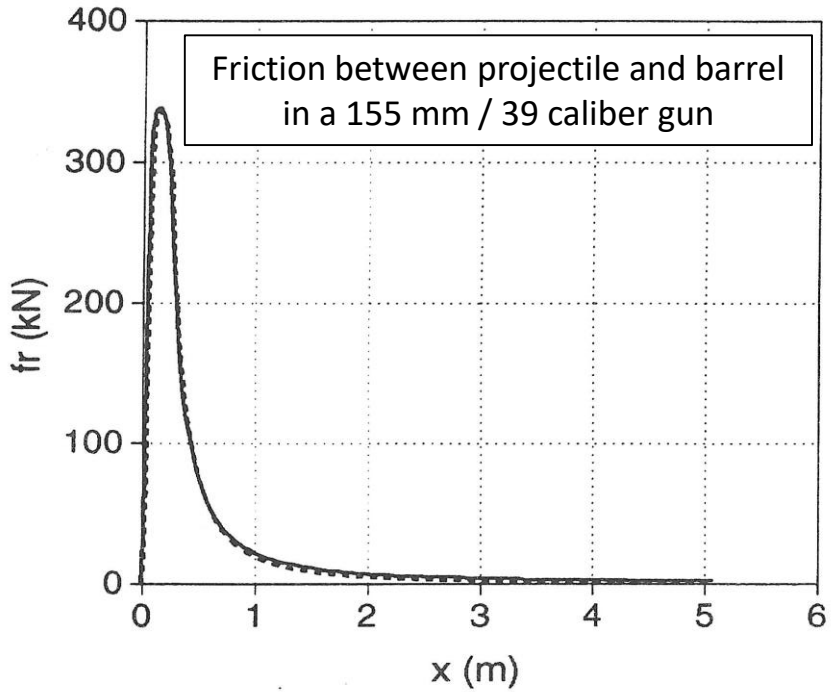


$\varepsilon\sigma T^4_{flame}$

$T^4 \sim 3800^4$

About 70% of radiation heating

Friction Between Projectile and Bore for 155mm Howitzer



A. Tuomainen "The Thermodynamical Model of Interior Ballistics," Acta Polytechnica Scandinavica, Applied Physics Series 205, Helsinki

This curve was integrated to estimate the energy using $q = \int F_f dx$. The result was115 kNm (85000 ft lb or 109 BTU) per shot. This 0.43% of the total energy in the propellant.

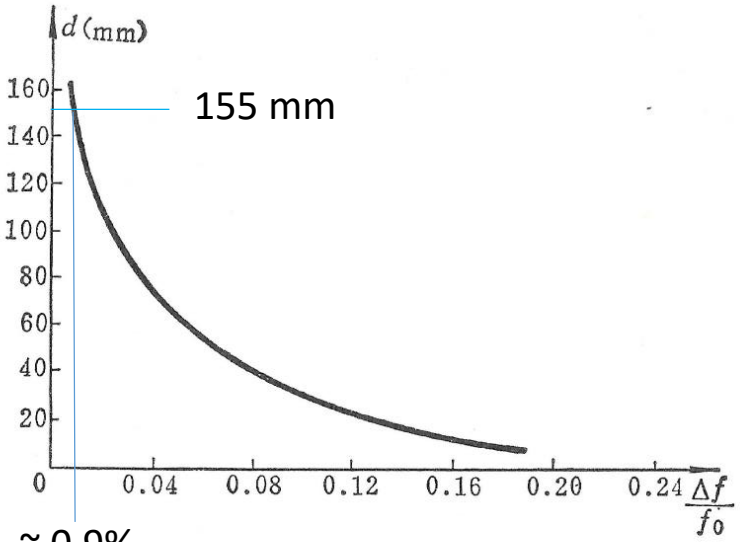


Fig 2.5 The heat loss $\frac{\Delta f}{f_0}$ vs the caliber of conventional guns

Bao Ting-Yu "The Theory of Potential Equilibrium for Ballistics and its Application," Defense Publishing House. Beijing, P.R. China, 1988.

The ratio of $E_{Friction}$ (Δf) to the total energy in the propellant, f_o, for guns of caliber 10 mm to 160 mm was calculated by Bao. The result of 0.4% is the same order of magnitude as in Tuomaninen.

Although the frictional energy is a small percentage of the total chemical energy available, it is a significant player in barrel heating

Barrel Heating During In-bore Period Summary

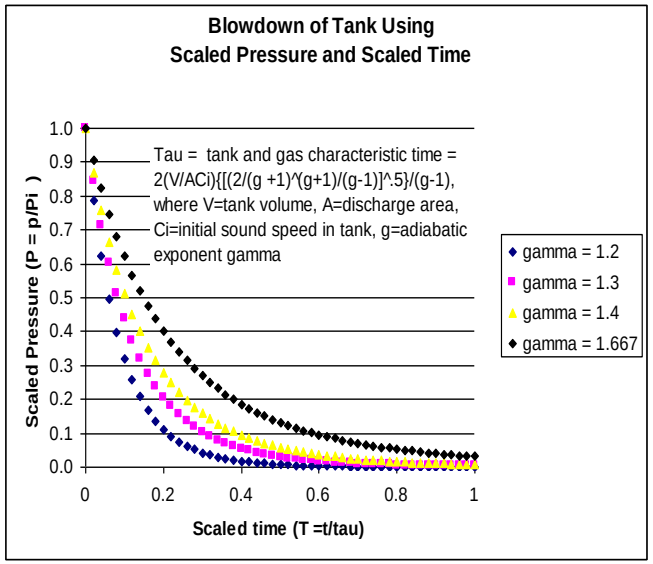
- Heating starts when the projectile first begins to move.

- The barrel heating is mainly radiation:
  Part of the radiation is from the propellant gas at the bulk temperature T_{bulk}
  Part of the radiation is from the flame temperature of the propellant T_{flame}

- The barrel heating by convection is small, and highest at the muzzle:
  Zero at the breech
  5% of the total heating at the muzzle

- The friction heating is hard to calculate and a small portion of the total energy, but a significant contributor to barrel heating.

Gas Dynamics Mechanism for Heat Transfer to Barrel During the Blow Down Time Period

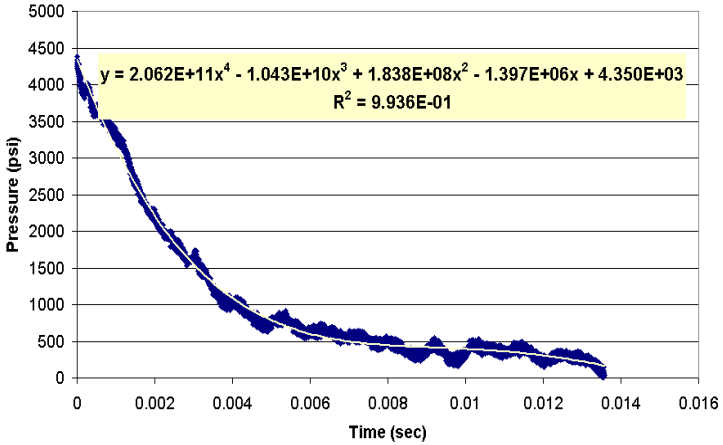


$$\left(\frac{p_f}{p_i}\right) = \left[1 + \frac{t}{\tau}\right]^{2\gamma/(1-\gamma)}$$

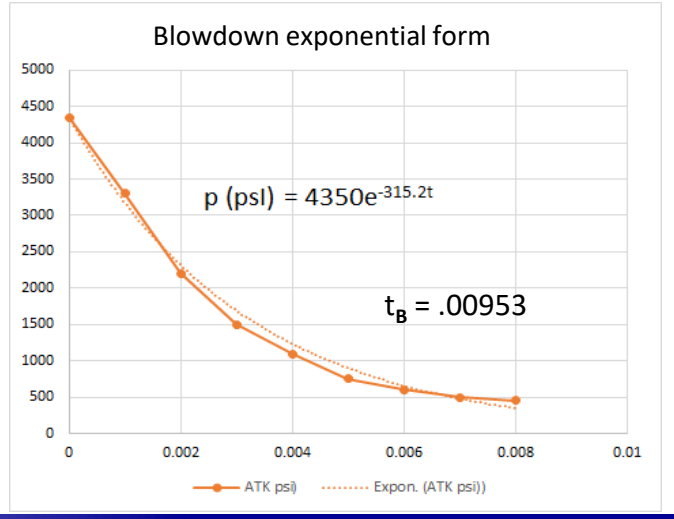
A =bore area
C = sound speed
p_i = initial pressure
p_f = pressure at time t/τ
τ = time constant
γ = adiabatic exponent

The velocity can be calculated from gas dynamics, then the Reynolds number, then the Nusselt number, and then the convection coefficient, h_c, and finally the heat flux q"

Jon J. Yagla "Pressure Vessel Discharge Non-dimensional Equations" Paper No. 19033551 28th International Ballistics Symposium, Atlanta



A pressure vs. time record from the muzzle in test. A curve was fit to the data.



Heat transfer shorthand:

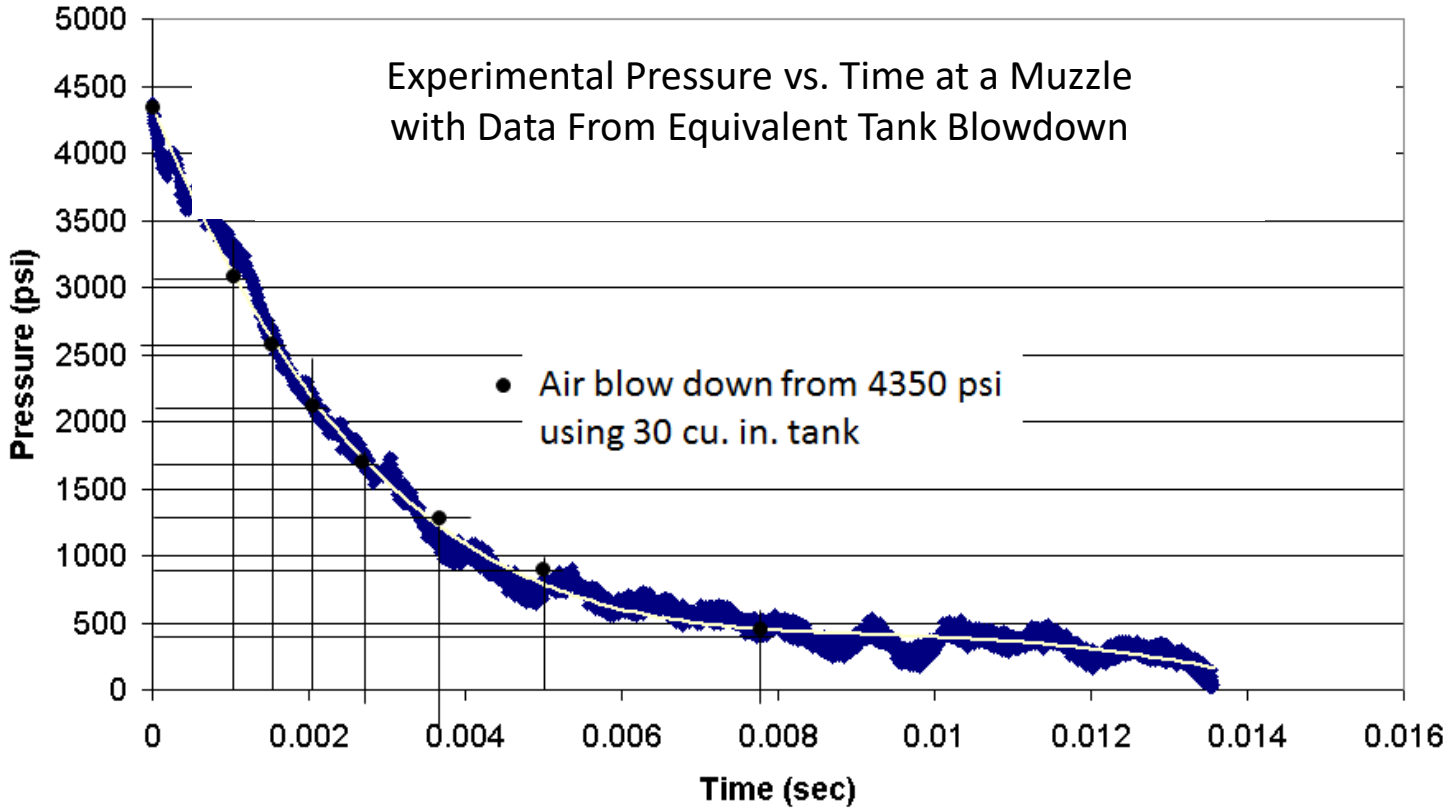
q = quantity of heat energy

Q = larger quantity, ∑ q's

q' = flow of heat per unit area, or per unit of time

q" = flow of heat per unit time per unit of area, the "heat flux"

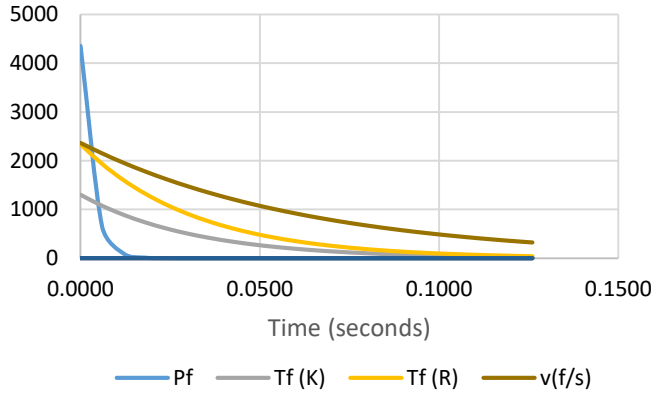
Matching Compressed Air to a Gun Barrel Blow Down



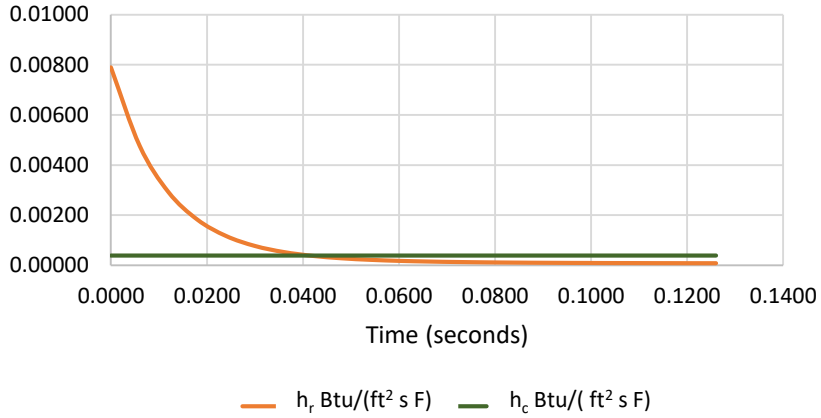
The compressed air blow down data are shown on the gun barrel blow down plot by the circular plotting symbols. They lie nicely on the gun barrel blow down curve. This shows a gun barrel obeys the dimensionless formulas. It also shows a cold gas discharge from a tank of compressed air can provide a precise simulation of the gun pressure. The formula is used to compute the temperature and velocity in the barrel, then Reynolds number, Nusselt number and heating from radiation and convection.

Example of Results for a 30mm Gun (work in progress)

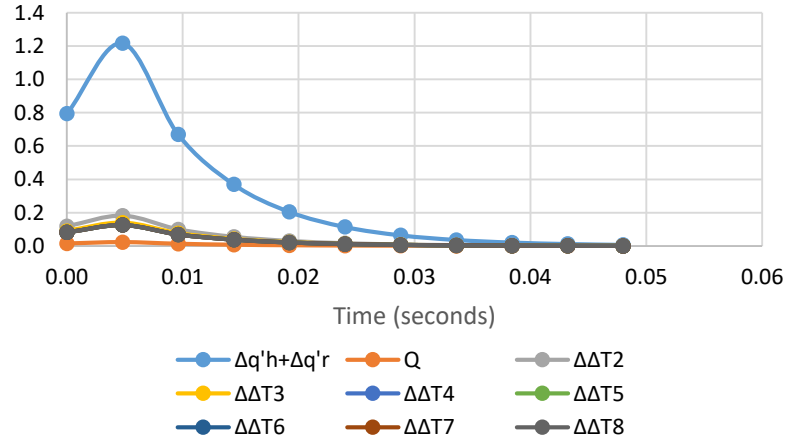
Blowdown Pressure, Temperature, Exit Velocity



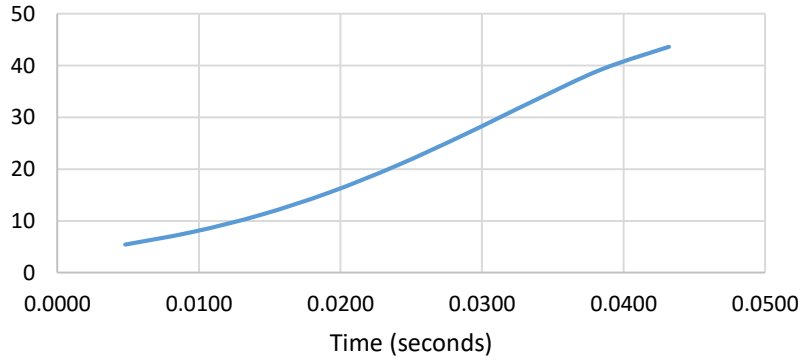
Heat Flux Coefficients h_r and h_c



Total Heat Flux and Zonal Differential Temperature Increase During Blowdown

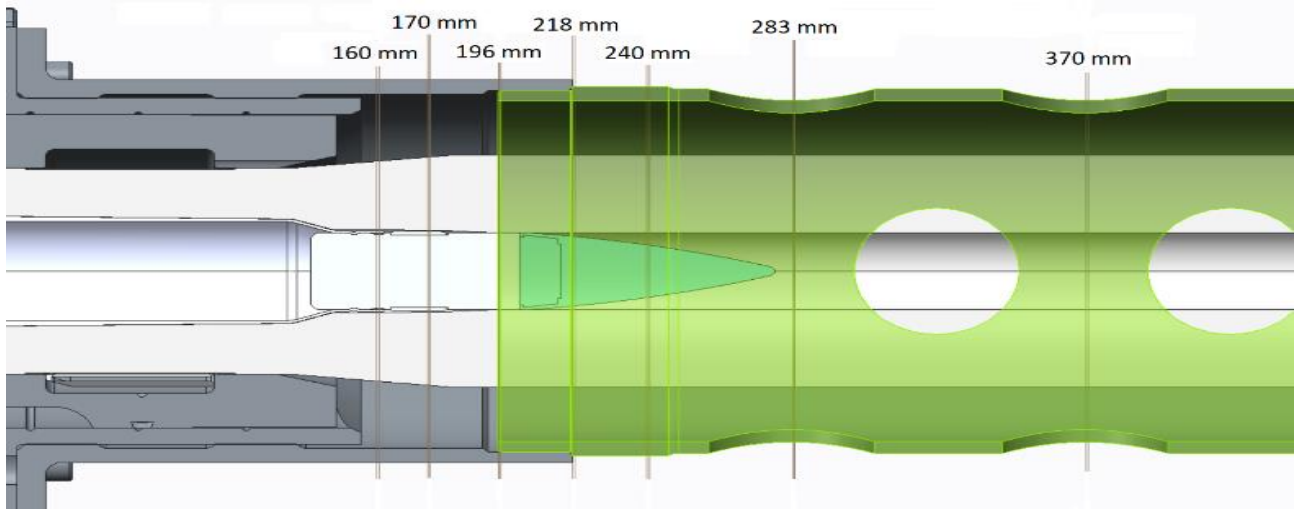


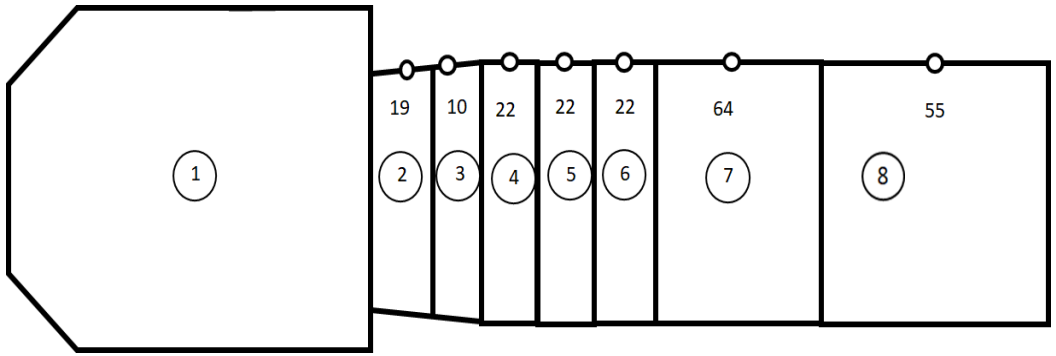
Instantaneous convection as a percentage of the total heating



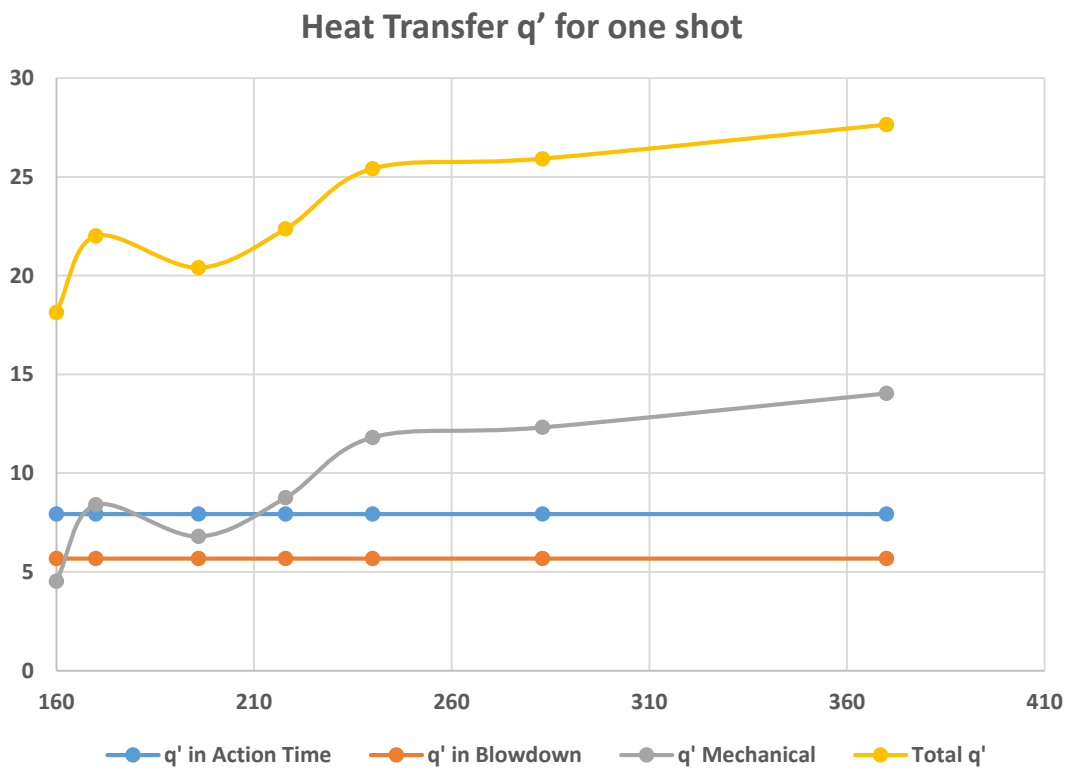
Lumped Parameter Model for Barrel Heating In Cartridge Region

The chamber region was divided into heating zones centered on thermocouple locations.
The volume, mass, surface area, and heat capacity was calculated tor each zone.
The measured temperature data were processed for each zone to determine q (per shot) and Q for a mission of 66 rounds.
Heating from radiation and convection was calculated for each zone.
The difference between the calculated heating and measured heating was calculated and attributed to mechanical losses of deformation and friction.





Semi-empirical Model Results for Chamber and Shot Start Region

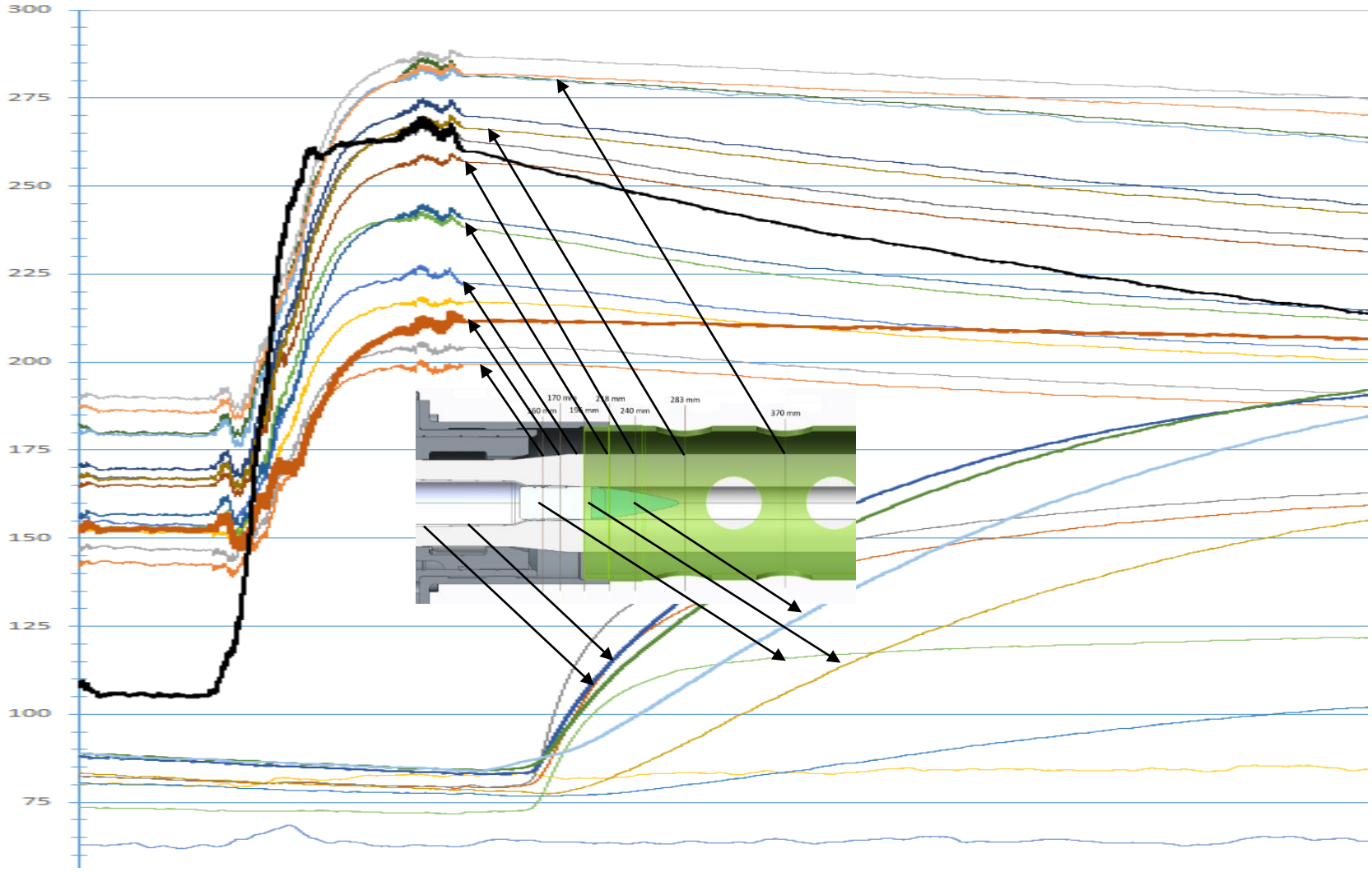


Using the yellow curve to left:

zone	x-station	q' in Action Time	q' in Blowdown	q' Mechanical	Total q'
2	160	7.93	5.68	4.53	18.14
3	170	7.93	5.68	8.40	22.01
4	196	7.93	5.68	6.80	20.41
5	218	7.93	5.68	8.75	22.36
6	240	7.93	5.68	11.81	25.42
7	283	7.93	5.68	12.32	25.93
8	370	7.93	5.68	14.03	27.64

- q' = total heat flowing into zone / inner (I.D.) surface area of zone (Btu/ft^2)
- $\Delta T = q' A / C$, where A = inner surface area and C heat capacity of the zone
- Heat consists of conduction, convection, and radiation. The mechanical portion is attributed to friction and other heating mechanisms

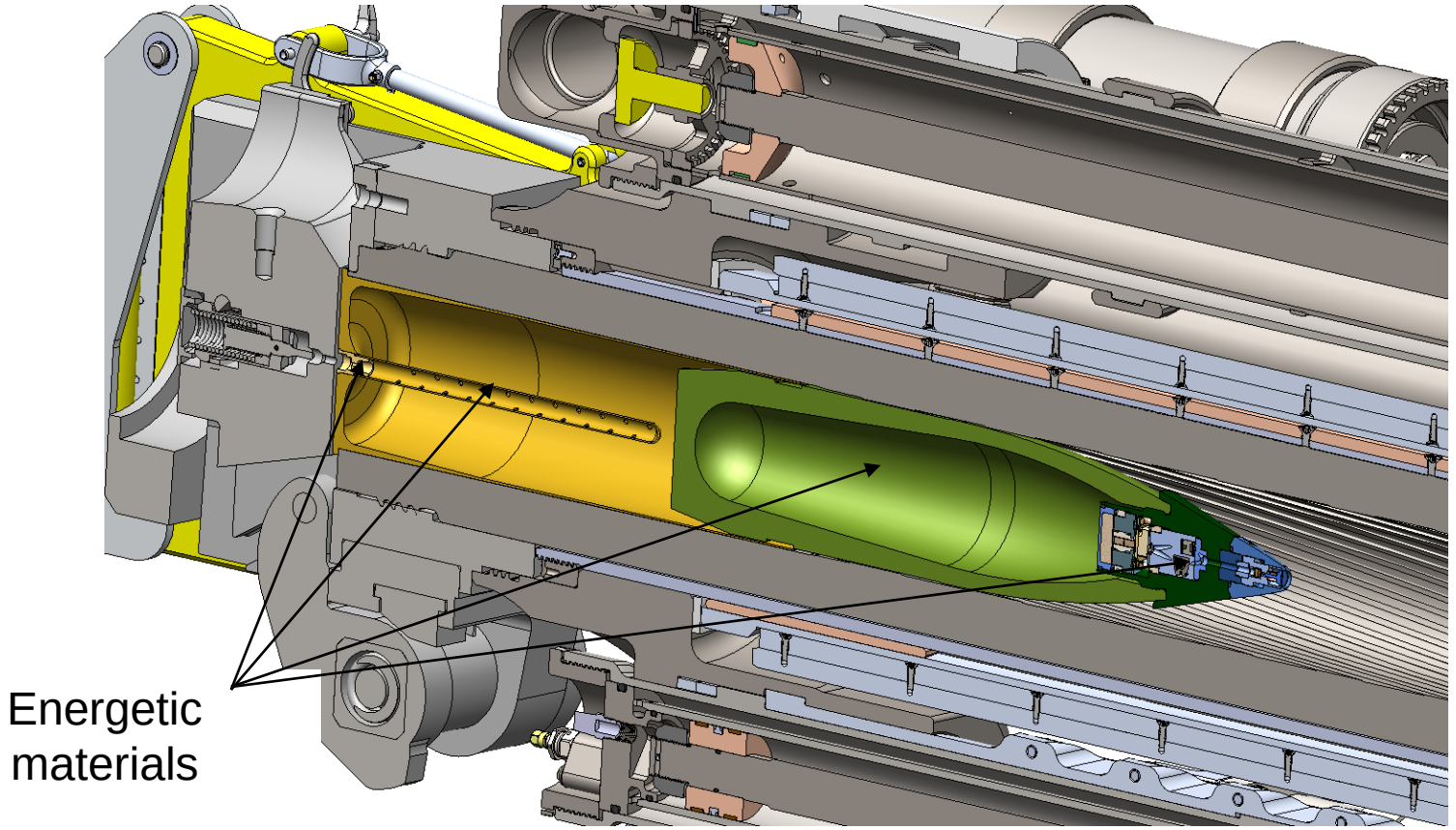
Example Cartridge Temperatures, 30 Rounds



Experimental Heating Data From 30 Rounds

x (thermocouple)	Tmax	ΔT
mm	F	F
160	202	142
170	214	154
196	227	167
218	243	183
240	268	208
283	272	212
370	286	226

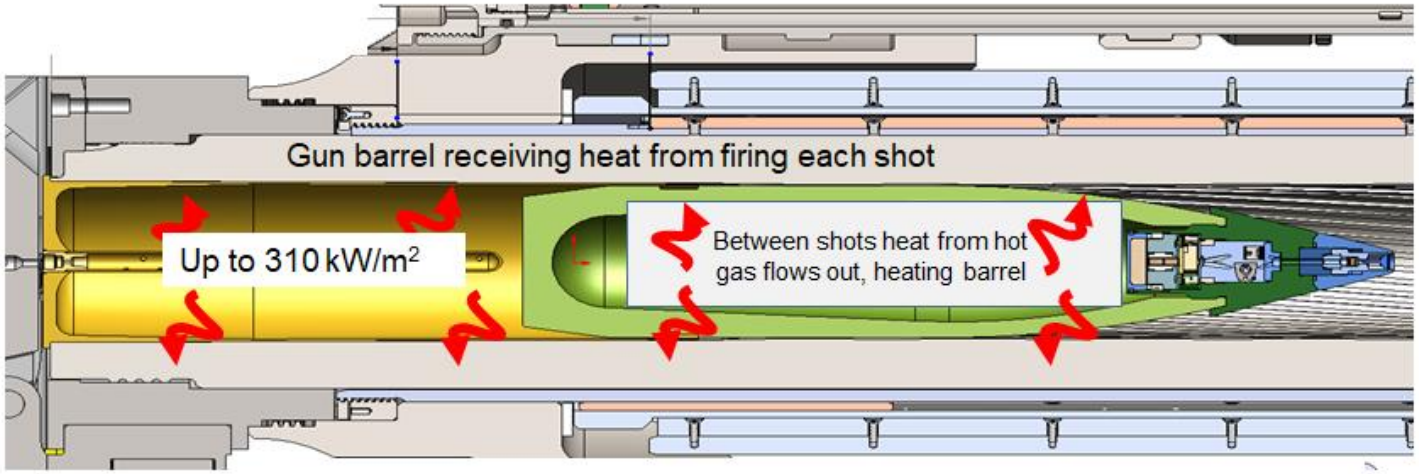
105 mm Hot Gun Cook-Off Experiment



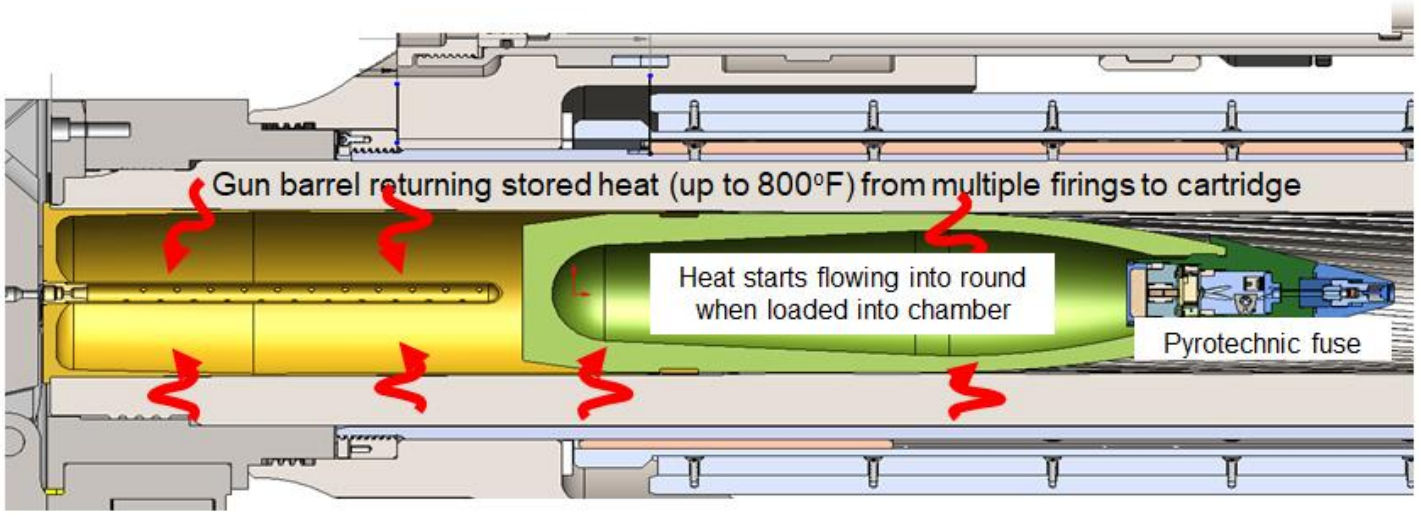
Repeated firing gets the gun barrel very hot. Should a round that has been loaded into the gun not fire, a potentially hazardous condition exists. ***If it not promptly removed it may cook-off.***

Heat Flows During Rapid Continuous Firing

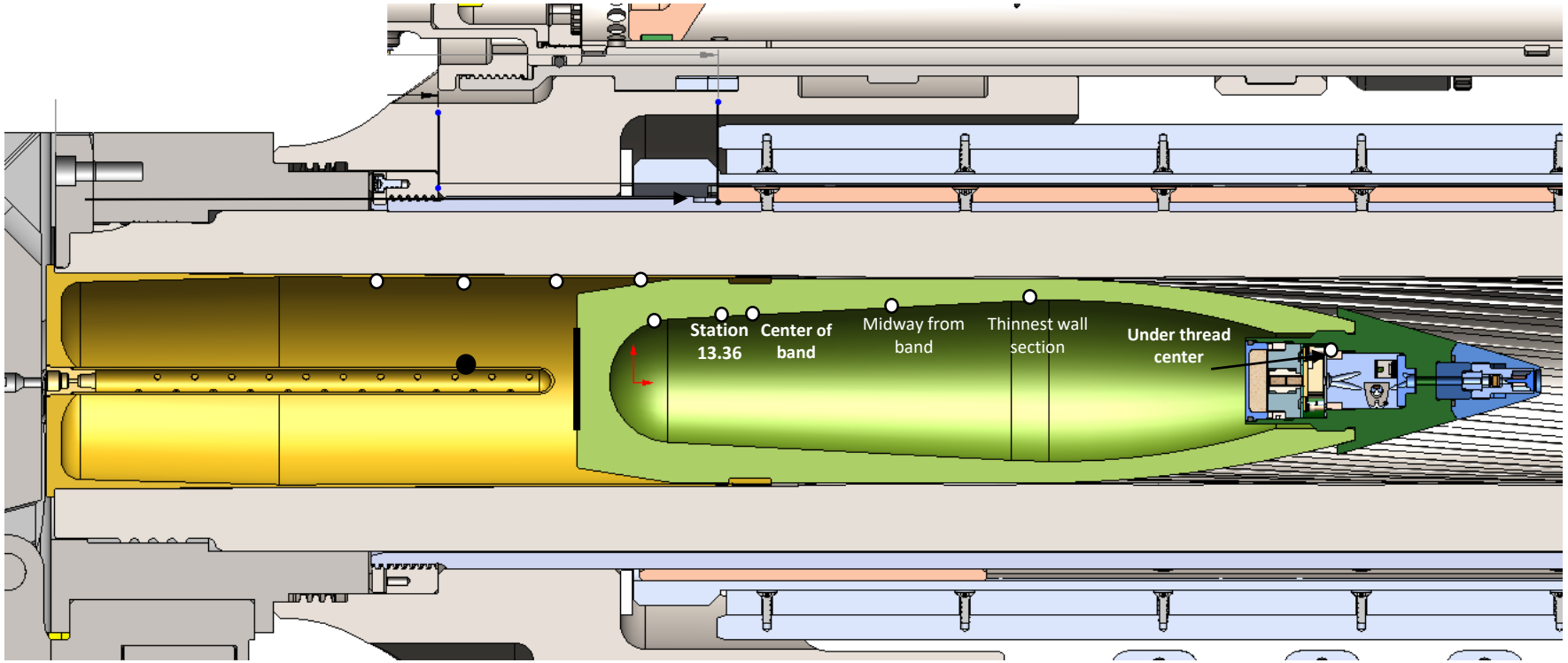
Heat flowing from chamber into barrel in $t_A + t_B$ (~ .020 sec)



Heat flowing from barrel into cartridge (~ 1 sec)



Opportunity to Leverage Data From Forthcoming Test with Instrumentation



105mm Hot Gun Test 2021. The projectile has been filled with material to simulate Composition B and the thermocouples have been tested.

Summary

Hot guns can be very dangerous

Barrel heating is in two stages: Action Time and Blow Down

Radiation, convection, and friction are the main heat transfer mechanisms

Radiation is caused by hot propellant gas and radiating propellant grains

Convection is relatively small, then increases during blowdown

Temperature measurements in the barrel and cartridge provide experimental confirmation and provide data to converge models